

# Chemical & Process Engineering

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## Work Study and Chemicals

ENTHUSIASM about work study in various parts of the world has led to a considerable flow of printed matter on the subject as related to industry in general. This the hard-pressed chemical manufacturer has searched eagerly, but often disappointedly, for anything that might be pertinent to the unique problems of his industry. In Britain, a notable exception is the proceedings of the Buxton Conference on work study that was organised by the Association of British Chemical Manufacturers in 1953, following the report of the Heavy Chemicals Productivity Team which toured the United States in 1952. These proceedings have done much to stimulate interest in the subject in the chemical industry, along with various information issued by the British Productivity Council.

Two booklets that have now been issued\* by the A.B.C.M. should do much to clarify the subject for chemical and other manufacturers. The first gives the essentials and includes detailed practical advice to firms on how to apply work study. Those who are apprehensive of the organisation and staff that work study might involve will be reassured by this booklet that a system can be built up gradually, from modest beginnings, as experience grows. To show that all this is not just theoretical, the second booklet (which most people will probably read first) records the actual improvements in efficiency and economy that have

been registered by 14 leading British manufacturers of chemicals and other products.

Thus, to take one example, Hardman & Holden Ltd. have applied work study at the design stage of new plants; improved layouts and reduced handling costs have resulted. The labour cost of one product (it is not stated which) was reduced by 50%, while the output of a plant was increased by 40% by a change in procedure, with an increase in labour force of 12% over the original. Various other work study successes of this company, who now propose to apply work-study techniques to their engineering department, are cited.

The work study team of A. Boake Roberts & Co. Ltd. have come to a very satisfactory arrangement with the company's chemical engineering and design staffs and, as a result, work study principles are introduced during the early stages of the design of new projects. This has resulted in improvements in design, particularly as to the operating controls and materials-handling arrangements of new plants.

Application of method study by the Eaglescliffe Chemical Co. Ltd. led to a simple arrangement for handling bags at a saving of £100 to £200 p.a. A major project for complete reorganisation of the packing and handling arrangements in one larger plant, with an estimated saving of £4,000 p.a., is also mentioned.

Imperial Chemical Industries' interest in work study is well known. In some factories it has been possible to make improvements in every department. Thus,

\*Notes on Work Study, Parts 1 and 2, 5s. (to non-members).

in a fertiliser factory, between 1949 and 1953, annual output was increased by 8% (without appreciable capital expenditure) and over this period the labour employed was reduced from 1,020 to 715.

But these are only a few examples. The diversity of the 14 companies in size, complexity and the goods they produce is best illustrated, perhaps, by naming them. They are, in addition to the companies already mentioned: B. B. Chemical Co. Ltd., Courtaulds Ltd., The Distillers Co. Ltd., Dunlop Rubber Co. Ltd., Thomas Hedley & Co. Ltd., Imperial Smelting Corporation Ltd., Kodak Ltd., Pilkington Bros. Ltd., Unilever Ltd. and the Wellcome Foundation Ltd.

It seems likely that many other manufacturers of chemicals and allied products will also prove the advantages of work study before long.

### **New vinyl acetate plants for Britain**

**T**HE growing importance of vinyl acetate in Britain is reflected in recent arrangements for the home manufacture of this raw material of the paint, adhesives and plastics industries. Earlier this year the British Oxygen Co. released details of its Durham facilities, which are manufacturing the stabilised monomer as well as polyvinyl acetate in granular form—both were previously imported. (We published a description of these plants in *CHEMICAL & PROCESS ENGINEERING* in March). It was only a few weeks afterwards that The Distillers Co. Ltd. and the Montreal company, Shawinigan Chemicals Ltd., announced the formation of a new company, Hedon Chemicals Ltd., which would manufacture vinyl acetate at Hull in Yorkshire.

The chemical is produced from acetic acid and acetylene, both derivatives of calcium carbide. Both the Durham and the Yorkshire vinyl acetate plants have independent supplies of carbide and acetylene to draw on.

Now British Celanese (see page 308) announce that they are erecting a plant for the production of vinyl acetate monomer. The company will use a process which they have developed themselves and, the chairman states in his annual review, they will be the first to operate it in Europe. Initially the plant will be capable of producing the equivalent of approximately one-third of present domestic consumption, and is so designed as to facilitate rapid expansion.

### **Canada's chemical output increases**

**S**OME inking of the range of Canada's chemical industry was given in our brief survey of the Canadian market for chemical plant and industrial chemicals last month. Production figures for 1954 that have now been released confirm the impression of a well-established and virile industry that keeps on growing. The value of chemicals produced in Canada during 1954 reached a record figure of some \$900 million. The 1953 figure was \$848 million. Exports in 1954 had a value of \$161 million, compared with \$138 million in 1953. Leading exports to European countries include synthetic rubber, polystyrene, penicillin and streptomycin, calcium carbide, dicyandiamide,

PVC acetate resins, acetylene and carbon black, synthetic iron oxide, pyrites, styrene monomer, plastics laminates, along with such new petro-chemicals as pentaerythritol, phenol, acetone, methanol, formaldehyde, acetic acid and acetic anhydride.

To meet demand from home and abroad, the Canadian chemical industry can draw on abundant domestic reserves of coal, oil, natural gas, salt, limestone, pyrites, woodpulp, and hydro-electric power. There are signs that both the heavy chemicals and the plastics sectors are heading for further rapid expansion in the immediate future.

### **Acetylene explosions in South Africa**

**A**N explosion which, during June, destroyed an acetylene generating plant at a Johannesburg engineering works, is the second serious happening of this kind in the Rand area, causing casualties and damage to property, in the past 12 months. There seems to be a need for South African regulations covering the generation and handling of acetylene, to be drawn up and enforced on an even stricter basis than in Britain in view of the danger to unskilled native workmen who cannot fully appreciate the risks but have been the first to suffer in these explosions.

Apart from normal precautions to avoid excessive temperature rise in carbide generators, plants of this nature in the Transvaal call for additional care in operation because of the low barometric pressure owing to the altitude of nearly 6,000 ft. and the rapid fluctuations in air temperature which occur, especially during the winter months when a rise of nearly 50°F. may be experienced in a few hours.

### **Nylon filter cloths**

**S**UCCESSFUL use of nylon filter cloths for rotary vacuum filtering of sewage sludge brought the West Kent Main Sewerage Board its reward for years of plodding investigations. The great saving of space that is now possible is even more of a boon than the saving in labour costs—two filters in an installation covering 100 sq. yd. can deal with more sewage than 57,000 sq. yd. of sludge beds. The long life of the nylon 1½-oz. nylon cloth (about 2,400 hr.) and the hourly yield of about 8 lb. of dry matter/sq.ft. make the process an eminently satisfactory one.

Although this achievement hardly seems recent enough to merit the headlines that a London newspaper accorded it last month, it is still very much a topic among sewage authorities as well as chemical engineers in other industries. At any rate, it earns a prominent place in a pleasing little booklet that comes from British Nylon Spinners Ltd. and which tells us, among many other things about the industrial uses of nylon, that nine-tenths of Britain's potteries now use nylon filter cloths, while a similar fabric helps solve the problem of disposing of sludge settled out of water discharged from coal washery plants after coal particles have been reclaimed. Again, at a Yorkshire sugar factory, the filter cloth bill has been halved, juice being filtered three times at about 90°C. at pH values between

## Comical Engineering Terms



"RIBBON MIXER"

11 and 7. All these and other claims for nylon filter cloths seem to be pretty convincing evidence that, if the filtering of sewage sludge is their latest major success, it will not be their last.

### Venezuela's chemical plans

THE Venezuelan Government is reported to be making good progress with its plans to set up plants for manufacturing nitrogen for fertilisers and explosives. In later stages of the development programme, plants for the production of chlorine, caustic soda and acetylene by-products will follow. Finally, the setting up of a petro-chemicals industry, based on the country's natural gas resources, is contemplated.

The formulation of these plans has been entrusted to the Petroquímica Nacional, according to a circular issued by the Board of Trade in London. This circular mentions the firms of Montecatini (Italy), Blaw-Knox (United States) and Uhde (Germany) as being involved in one connection or another where the chemical and fertiliser plants are concerned. Invitations to tender for the various items of equipment required will be issued privately to selected manufacturers.

In the first stage, a number of units, various capacities, will be required for the production of anhydrous ammonia, nitric acid, ammonium nitrate, sulphuric acid, sulphate of ammonia, superphosphates and urea. It has been decided to set up the plants near Moron in the region of Puerto Cabello.

The plans for the petro-chemicals industry open up the prospects of manufacturing plastics, synthetic rubber, detergents, solvents and a number of other chemicals in Venezuela.

## Whither leather?

WITH every new plastics and synthetic rubber development, the position of the world's leather industries grows less comfortable. It is plain that existing leather processes must be greatly improved upon or even replaced by new ones if this time-honoured industry is to survive.

The need to intensify research, particularly chemical research, in this field is gradually being recognised by the industry's leaders. Chemical engineering, which is involved in the tanning processes, will also have an important part to play in effecting improvements.

So far, however, there is little sign of any concerted efforts being made to solve the problems, perhaps because in many countries the industry is able to stand its ground for the moment (*e.g.* in Finland, as reported on another page) and many leather manufacturers are inclined to be complacent.

What is wanted is more promising new developments such as the solvent tannage process, which was recently commented on by Mr. E. S. Flinn, president of the American Leather Chemists' Association. The idea of this process originated in Germany before the first world war; further work was done about 1930 by the U.S. Tanners Council Research Laboratory, and now it is being tried out on pilot-plant scale. This process is one example of a valuable research contribution; surely chemistry can provide the leather industry with many more such.

### King Solomon's mineral resources

IN contrast to the gloomy outlook in Israel-Egyptian relations are bright reports from old Palestine of new industries out to develop vast mineral resources. Among the first group of ancient resources now utilised to serve modern industry is copper, one of the new extraction plants being sited at Timmah, in the Wadi Menaiah, where Solomon won copper ore for smelting at Ezion Geber. Potash from ancient Sodom gives way to potash from the Dead Sea, an 'almost inexhaustible' source of potash as well as of bromine. Whereas the Dead Sea yielded at one time only salt and asphalt, there are magnesium salts in abundance, a point perhaps anticipated for a country which had a Dr. Magnes as head of its university! The Arab destruction of one Dead Sea factory was compensated by a fresh start at the southern end works of the Palestine potash concern.

Apart from potash, magnesium and bromine, the new reports tell of vast deposits of other minerals. A 100 million tons of rock phosphate in the Great Crater of Maktesh Hamadol ensure that Haifa, with its superphosphate industry and a promised synthetic ammonia plant, will be noted for riches other than the oil-refining that has been so much in the news within recent years. There are extensive gypsum deposits in the Maktesh Ramon region with the first recovery plant, while felspar and white silicate sands are further resources as yet awaiting development.

Ancient olive oil formerly supplied the needs of a soap industry which has now so grown as to require



imports of copra, palm oil and sunflower oil to maintain production. The Shemen oil and soap works form part of the Hamashbir Hamerkazi or Central Wholesale Supply Co-operative, a concern which also controls the Hamegabbar Rubber Co., Hatzamar Wool Processing, and Fertilisers & Chemicals Ltd. A second corporation, the Solel Boneh, controls the Kharsa ceramic works, the Vulcan foundries, the Nesher cement works and the Phoenicia glass works.

Textiles, leather and plastics industries have come to modern Israel as have manufacture of pharmaceuticals, dyes, paints and waxes. While the diamond-cutting industry came as a result of the German occupation of Holland, other industrial workers from Poland and Central Europe have likewise benefited the chemical and allied industries. It is a far cry from King Solomon's appreciation of the mineral resources of this ancient country to the new industries now springing up; much of the latter depending in some way on guidance from the Weizmann Institute of Science at Rehovoth, an appropriate memorial to that great chemist who won such eminence with his fermentation process for acetone during his spell at Manchester University.

### Germanium from boilers

THE world's germanium supplies are being eaten up rapidly by new applications in electronics, and there is a constant search for ways of extracting the metal from low-grade sources. Possible sources range from zinc blends to waste wood products. Coal is earning a major share of attention at the moment, attempts being made in various parts of the world to extract germanium from the products of carbonisation or combustion, such as flue dust, coke, tar and ammonia liquor.

In the United Kingdom, as reported in this issue, attention has been given to the waste products that are deposited in the flues of a coke-burning gas producer. But at present it seems very likely that important quantities of the germanium pass from the flues in the form of a very fine dust which can be carried through the chimney stack without deposition. In fact, samples of such dust have been collected and have been shown to contain about 1% of germanium.

Elsewhere in the U.K., there are promising signs that germanium could be extracted from boilers used in electricity generating stations. Examination of the deposits and grits from various parts of such boilers led to the theory that the element might be passing through the boilers in a volatilised state. The distribution of germanium between the clinker, coarse dust and fine dust in retort, chain-grate and pulverised-fuel-fired boilers was examined and the results were briefly discussed by Mr. E. A. Howes and Mr. B. Lees in the *Journal of the Institute of Fuel* in June. Some 30 to 40% of the germanium in the coal is present in the fine dust from mechanical-stoker plant in concentrations suitable for commercial extraction; concentrations may be enhanced by fuel selection. The remaining 60 to 70% of the germanium is present in the clinker and coarse

dust, in concentrations which are not commercially recoverable.

These experiments suggest that over the range of fuel-bed temperatures and boiler size examined, the quantity of germanium in the fine dust is not appreciably affected. There appears to be no method of extracting germanium from the dust from pulverised-fuel-fired boilers, owing to the dilution effect of the fine pulverised-fuel ash, which would be economically feasible.

It was decided that the investigation should be taken further, to the stage of commercial prototype pilot units and, in collaboration with manufacturers, work is proceeding.

### Moisture-proofing cold plant

RUBBERISED bitumen, used as a moisture-proof covering for the insulation on refrigerated plant, is claimed to have a number of advantages over the bitumen emulsions and various solvent-bound bitumens that have been used previously for this purpose. Brittleness, cracking by shrinkage, degeneration through weathering and a variable degree of permeability to moisture are among the important defects that have been encountered with previous coatings. Rubber-modified bitumens, or R.M.B., as the new materials are called, have been in use at the Billingham works of I.C.I. Ltd. for two years and, it is stated, are standing up to atmospheric conditions satisfactorily. Breakdown has occurred only where there has been continued immersion in water.

Degeneration during storage has been experienced, but it is thought that this difficulty has been overcome with the latest types. They cannot be applied by brush but at least one type can be diluted with water and sprayed. Further work will be carried out on this method of application.

Although inflammable, R.M.B. does not burn so readily as bitumen, it is stated; nor does it melt, drip or spread fire. The chemical resistance of the material is such that, briefly, it can be said that the chemicals that attack natural rubber will attack R.M.B. It should be able to withstand acid splashes of most concentrations, except concentrated nitric acid. It is particularly susceptible to attack from splashes of solvents and should be protected. Because it is based on an emulsion it will not withstand continued immersion in water, but a coat of bituminous aluminium paint appreciably reduces attack.

Describing the new material and its uses in a recent technical bulletin of the British Rubber Development Board, C. J. Moss and P. Stevenson state that the present cost of the R.M.B. is about 1s. per lb. This is about twice that of an ordinary emulsified bitumen by weight, but, it is claimed that, when considered from the point of view of an equal vapour seal, it is much cheaper. Variations in the rubber content to reduce price and/or to achieve different properties (e.g. jointing materials) is a possible line for future work.

The sprayable emulsions may have some useful applications in the future and more work is needed.



# POLYTETRAFLUORETHYLENE

## in the Chemical and Allied Industries

By E. Franks, B.Sc., A.M.I.CHEM.E.

*One of the newer plastics that have been added to the chemical engineer's armoury of corrosion-resistant materials of construction is polytetrafluorethylene. It can now be used as extruded tube and-rod, lip rings and seals for pumps, valve packings, joints, etc., while promising innovations include PTFE dispersions and PTFE bonded to rubber, metal or glass fibre. Here is a survey of these and other developments in Britain and the United States.*

**POLYTETRAFLUORETHYLENE** (PTFE) is a fully fluorinated ethylene polymer which is finding many uses owing to its outstanding properties. The working temperature range of  $-100^{\circ}\text{C.}$  to  $+250^{\circ}\text{C.}$  may be exceeded in certain circumstances. It is fully resistant to the action of solvents and other chemicals, apart from fluorine and molten alkali metals. The material also has a low coefficient of friction. It is non-wetting and non-adhering. Finally, the electrical properties are similar to those of polythene with the added advantage of the wide temperature range.

The polymer was first developed in the United States by Du Pont under the name of *Teflon* and information about various products has appeared in American literature since 1946. In Britain, I.C.I. Ltd. produced *Fluon*, as they named the material, on a pilot-plant scale towards the end of 1947. Owing to the poor flow characteristics and lack of true melting points the polymer is difficult to process. The best results are obtained from simple mouldings, and the technique of moulding followed by sintering is comparable to that employed in powder metallurgy. From these blocks, tape can be veneered, diaphragms can be knifed off and the material can be readily machined into any desired shape.

Although new techniques in processing and new types of dispersions are enlarging the scope of the polymer, the difficulties in normal processing have tended to limit the use of PTFE. In the chemical field, its corrosion resistance and low friction properties are mainly employed in the form of joints, diaphragms and packings.

### Packings for centrifugal pumps

To obtain the best results from PTFE packings in centrifugal pumps, various factors have to be considered



U-rings moulded from PTFE.

[Crane Packing Ltd.]

carefully. In all cases the pump shaft needs to have a good finish to prevent scoring of the plastic, and the alignment should be correct within 0.0001 in. As it is a very efficient insulator, it is essential that the material be used so that local heating is minimised by reducing friction or by cooling. If heating-up in the bore is produced at high running speeds, it is possible to cause breakdown of the PTFE. The local heating gives rise to expansion, which produces a greater bearing load, thus aggravating the condition and leading to a rapid rise in temperature. This cumulative effect can cause eventual failure of the PTFE owing to shear, and small particles are torn off. To increase service life, the packing must be bedded-in very carefully; ample cooling should be applied and,

if possible, a fluid or grease film should be kept between the packing and shaft.

In general, packing rings of solid PTFE are not very satisfactory when used alone on centrifugal pumps, as the material is too hard to allow ready adjustment by pulling up on the gland. The consequent close dimensions required for effective sealing can lead to local heating. Although constant grease lubrication through a lantern will allow this type of packing to be run successfully, solid rings are more usually employed as header rings in conjunction with other types of PTFE packings.

The best results have been obtained from a bulk material manufactured from PTFE shreds, graphite and a resistant lubricant. This type of packing readily moulds itself around the

shaft. The lubricant present helps both to seal any interstices and to provide a film between packing and the shaft. Thus frictional heat is kept to a minimum, the gland can be readily adjusted and the corrosion-resistant properties of the plastic are used to the full.

Other packings used on centrifugal pumps consist of a wrapped or braided PTFE jacket on a resistant core, e.g. blue asbestos rope or braided glass yarn. These rings are resilient and the life obtained is dependent on running conditions, as failure is due to mechanical wear of the PTFE jacket. The wrapped rings with the larger contact area tend to wear more readily than the braided type and both styles are improved by the use of  $\frac{1}{16}$ -in.-thick PTFE spacers between the rings. This helps to seal back leaks and increases the labyrinth effect.

In Britain, various types of PTFE packings for use on centrifugal pumps handling difficult chemicals have been made available by Crane Packing Ltd., of Slough, during the past few years. The following examples indicate the life to be expected from these corrosion-resistant packings:

(1) Braided packing with spacers has proved satisfactory on a pump handling molten naphthalene and caustic solution. The service conditions of 4 hr./day followed by steaming-out gave, with other packings, a life of 4 to 7 days before leakage of naphthalene became excessive. The PTFE packing increased this service to 2 to 3 months.

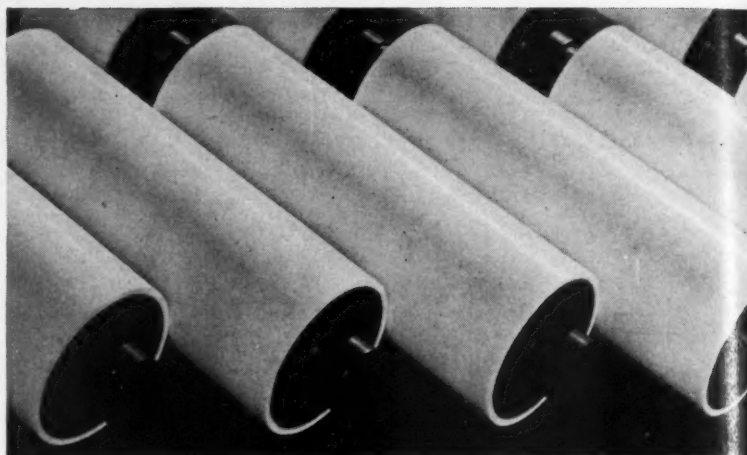
(2) Various packings were tested on a pump handling a viscous dope in methylene chloride (shaft  $2\frac{1}{2}$  in. at 60 r.p.m.). The results obtained were:

Packing	Life (hr.)
(a) Wrapped packing on blue asbestos core .. ..	225
(b) Braided packing on blue asbestos core .. ..	700
(c) Solid rings mica lubricated ..	—*
(d) Alternate solid and braided rings .. ..	500
(e) Alternate solid and braided rings, but lantern added without and lubricant injected ..	3,000 failure

\*Test abandoned after 175 hr. Adjustment difficult.

(3) A British LaBour pump handling concentrated sulphuric acid at ambient temperatures (shaft  $\frac{7}{8}$  in., speed 2,800 r.p.m.) was packed with the shredded type of PTFE packing. This material gave 1 year 7 months' trouble-free service prior to removal on overhauling the pump.

Mechanical seals are now widely used on rotary shafts. Many equipment manufacturers, particularly those



[Edison Swan Electric Co. Ltd.]

The anti-adhesive properties of PTFE make it useful for rollers handling sticky materials. The rollers seen here, fitted with PTFE sleeveings, are for bakery use.

serving the petroleum and chemical industries, are incorporating this method of sealing, as it ensures long periods of trouble-free running without leakage. These face seals normally contain rubber 'O' rings or bellows and, despite the large range of synthetic rubbers, this limits the services and temperature for which they are satisfactory. By replacing the rubber members with PTFE wedge rings, lip seals and joints, many of the difficulties are overcome.

#### Packings for reciprocating pumps

With reciprocating pumps the rubbing speed between shaft and packing is low and the problem of build-up of frictional heat is not severe. Thus full use can be made of the resistance to attack and low frictional properties of PTFE. Starting with a pump in a good mechanical condition, i.e. a good finish to the rod and perfect alignment, PTFE packings of various types can provide an increased service life without shaft wear. Moulded rings of shredded material, graphite lubricated, have given over two years' service on a pump handling cresol, sulphuric acid and other chemicals. E.C.D. injection pumps have been packed successfully with solid header rings combined with shredded and lubricated bulk material. This combination has operated at pressures up to 2,000 p.s.i.

Recently, lip-type moulded PTFE rings have been employed on high-pressure pumps. A set used on a Howard injection pump handling *p*-chloro-nitro-benzene at 3,000 p.s.i. and 270°F. has operated successfully for 300 hr., 150 hr. of which have been continuous service.

#### Valve packings, joints and gaskets

Solid rings, jacketed packings and other PTFE packings have been used successfully on valve work, where the corrosion resistance of the material ensures a long packing life. In addition, easy adjustment is assured and danger of the valve spindle sticking is virtually eliminated. Square-section moulded rings have been used on valves handling a variety of chemicals, and rings are also in use in valves on oxygen cylinders and similar services.

A set of wrapped rings with a PTFE cord core have operated at a pressure of 200 p.s.i. and temperature of -150°C. The packing at this temperature was still sufficiently resilient to allow gland adjustment. A test using liquid oxygen at -183°C. indicated that the PTFE still retained a measure of resilience and flexibility after immersion.

The United States have adopted lip-type packings for general use in control valves operating at temperatures up to 250°C. This type of packing is now being supplied to valve manufacturers in Britain.

A test set of three rings has given 10,000 reversals in a control valve without leakage or sticking. The rings are also ideal on arduous services where leakage or sticking must be avoided. Initial tests on chlorine cylinder valves have proved them to be satisfactory. This type of ring has also worked in valves at 3,000 p.s.i. for six months without any trouble.

**Joints and gaskets.** For normal pressures, PTFE joint rings of  $\frac{1}{16}$  to  $\frac{1}{4}$ -in. thick give effective sealing. Care must be taken that the flange bolts are not tightened unduly, as this can cause

flow of the PTFE and failure of the joint. To obtain additional sealing, e.g. for vacuum work, the joint ring can be ridged or the flange can be grooved.

At high pressures, PTFE tends to extrude. Joint rings can still be employed by fully containing the ring, e.g. between tongue and grooved faces. In this way PTFE joints have been used at pressures up to 3,000 p.s.i.

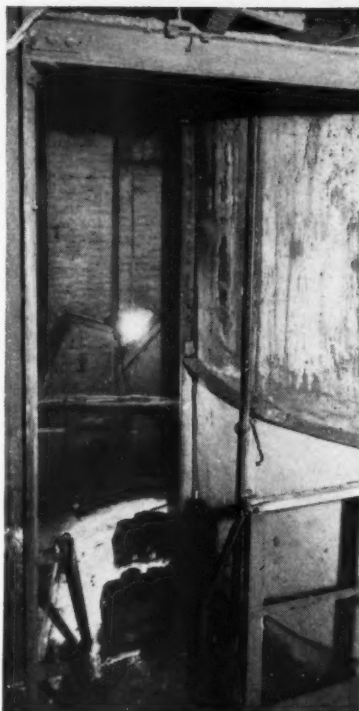
A large number of PTFE joints are used on glass pipe assemblies. The type of gasket commonly employed is in the form of a radially split ring. This forms an envelope around the standard joint and the glass pipe seals on the PTFE. Thus complete protection is obtained in the bore and of the faces of the standard joint. The most satisfactory insert material is compressed asbestos fibre, which allows the PTFE to be trapped between it and the glass. A soft insert can allow the PTFE gasket to be pulled down excessively on the outer diameter, thereby splitting the envelope in the bore.

#### Bearings, bushes and sealing rings

Having a very low coefficient of friction, PTFE is used where self-lubricating properties are required. Again it is essential to prevent local heating and to ensure that the bearing load is not too high. Consequently, bushes of solid PTFE have mainly been employed as self-lubricating bearings on instrument work and similar light duties. However, bearings capable of running under dry conditions have been manufactured from metal bushes prepared with a surface coating of PTFE. By this means the frictional heat developed is readily conducted away and, in addition, the plastic is fully supported by the metal.

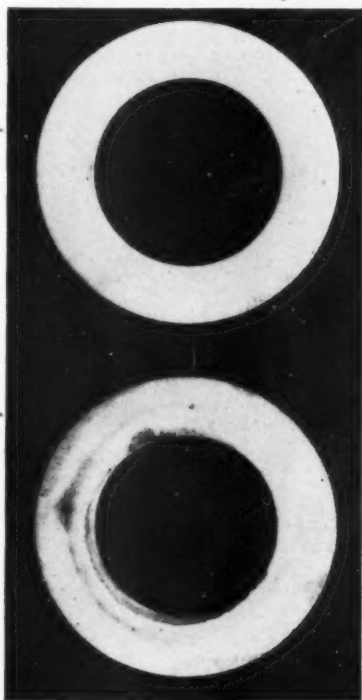
Gland bushes machined from PTFE have been used on aluminium valves handling concentrated hydrogen peroxide. They replaced aluminium bushes which caused trouble due to binding. Similarly, difficulties experienced with seizing between the plug and body of a stainless-steel cock were eliminated by use of a PTFE sleeve on the plug. The cock was handling concentrated nitric acid.

The self-lubricating properties of PTFE have been employed in cylinders operating on dry air or other gases. Lip rings and spring-loaded piston rings have been used to replace conventional rubber or leather rings which do not work satisfactorily under the dry conditions. Rings of PTFE are also capable of working over a greater temperature range.



[I.C.I. Plastics Division]

Above: Sulphuric acid plant of Hardman & Holden Ltd., Manchester, in which 'Fluon' (PTFE) packing rings are used at pipe joints from drain boxes at foot of tower. Below: An unused 'Fluon' ring and one after two years' service in the Hardman & Holden plant.



#### Tape and sheet PTFE

Continuous lengths of tape and sheet up to 6-in. wide are obtained by veneering from blocks of suitable widths. A great deal of this material is used for electrical insulation purposes owing to the excellent dielectric properties of PTFE. Apart from using .005 to .010-in. tape for wrapping on wire, valve holders have been stamped from thick tape (.030 in.). The low power factor over a large frequency range (50 to  $10^9$  cycles) makes PTFE particularly suitable for ultra-high-frequency work.

Diaphragms can be made by cutting from sheet or parting from a thick disc. To obtain maximum strength and flexibility the material is further processed by heating to the transition temperature of 327°C. and rolling. PTFE diaphragms have been used in control valves. For maximum flexibility the thickness should not exceed .020 in. and the best results are obtained by employing a composite diaphragm made of layers of .003 to .010 in. thick. Wherever possible the diaphragm should be supported to prevent undue stretching, as PTFE tends to be plastic rather than elastic and has a slow recovery. The material is very effectively used as a resistant barrier in conjunction with metal and rubber diaphragms.

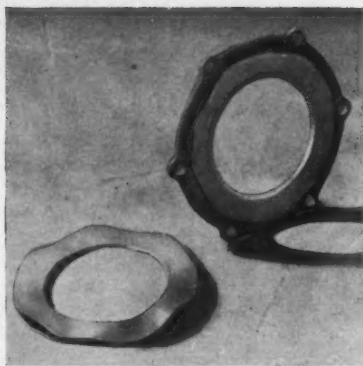
The anti-adhesive properties of PTFE are employed in the packaging trades. Tape, heat treated to give additional heat stability, is used as an anti-stick barrier when heat sealing Cellophane, polythene, waxed paper, etc.

#### PTFE dispersions

Dispersions of PTFE have been employed in the United States. In Britain, only a small amount of work has been carried out, owing to supply difficulties. The dispersion usually contains 50 to 70% finely divided polymer in an aqueous solution, a suitable wetting agent being added. Primer coats contain acid to give an etched key on metals. The material is sprayed on to the surfaces to be coated, dried and then the whole is baked at 400°C. A coat of approximately .0005 in. is put on at each stage and the above procedure is repeated for each coat. The lack of melting point and the presence of the wetting agent tends to prevent a 100% guaranteed coverage without pinholes. Consequently, dispersions have mainly been used to obtain non-stick surfaces on extrusion screws, packaging machinery, dough rolls, etc.

However, other processes employing





[Crane Packing Ltd.]

A gasket shield of PTFE showing, on the right, how a disc of jointing material is inserted between the leaves.

dispersions should prove of interest in the future. The colloidal particles can be coagulated and then redispersed in a suitable organic lubricant (e.g.  $C_{16}$  to  $C_{20}$  hydrocarbons) to form a paste. This material can then be readily extruded through shaped nozzles to give tapes or thin-walled cylinders. After extrusion the lubricant, of which approximately 20% is used, is removed by suitable solvents or volatilisation and the polymer articles are then sintered to form the finished product.

#### Some recent developments

Rods and cylinders of a few feet in length and varying diameters are now being manufactured in the United Kingdom by both Crane Packing Ltd. and Edison Swan Electric Co. The latter company are hoping to produce corrosion-resistant piping of varying wall thickness. By use of the normal polymer, cylinders with a minimum wall thickness of  $\frac{3}{16}$  in. will be manufactured. However, by employing a very fine polymer (particle size  $10\mu$  approximately), it is hoped to obtain a range of cylinders with thickness varying between 0.010 and  $\frac{3}{16}$  in. Other developments include thin-walled sleeves for dough rolls and laboratory beakers of various shapes. It is stated that the beakers are corrosion resistant, give freedom from contamination and can be used up to temperature of about 300°C. This allows heating of solutions on steam baths and controlled hot plates.

In certain cases the anti-adhesive properties of PTFE are a disadvantage. Mention has already been made of the use of a polymer film on metal surfaces for lubrication purposes. This can be done by pressing thin PTFE sheet into a porous metal under high pressures and at a temperature of about

360°C. A method has been developed of building up a layer of metal on PTFE, which can be applied to sheet, diaphragms, cylinders, etc. At present the method is established for non-ferrous metals. However, experiments are being carried out on the use of ferrous metals, including stainless steels.

The metallised PTFE can be bonded to other materials by conventional means, providing the temperature is kept below that causing breakdown of the polymer. Thus joints to metals can be made by soft soldering or use of resin cements. A completely bonded surface can be produced between a PTFE and rubber diaphragm, using a metal and rubber vulcanising technique. This composite material can then be used in diaphragm valves and diaphragm pumps. By this means the resilience of the rubber is combined with a high measure of corrosion resistance.

#### PTFE-bonded glass fibre

The use of glass fibre as an inert filler is well established in the plastics industry. Very thin glass tapes impregnated and coated with PTFE have been used in the United States for barriers in heat-sealing machines and for electrical insulation purposes. The glass imparts increased strength without adversely affecting the properties of the polymer. This material is now being manufactured in Britain.

Experience gained in the manufacture and use of PTFE products over the last few years has already indicated that the material, if correctly applied, can help to solve many difficult problems. The long service life, which can be obtained with the minimum of maintenance and overhaul, should amply repay the initial expense of the PTFE. For the future, the polymer has such a wide range of properties that new uses are bound to arise.

### Industrial Projects in Bolivia

Among industries for which private investment is being encouraged under Bolivia's industrial development plans are the following:

**Caustic soda.** Salt is abundant and, when hydroelectric power has been developed, could be used to manufacture caustic soda, which is now imported. The market for this product would not only be the small local industries but also the much larger industries in southern Peru and northern Argentina. As the supply of cheap power becomes adequate, this

### BURSTING DISCS

and their use on process plant

Next month, Dr. J. M. Pirie writes for us on bursting discs—what they are; what they do; what types are available; advantages and limitations; practical applications; materials of construction; compound assemblies; special applications; and future prospects. The whole will be abundantly illustrated with photographs showing a variety of bursting-disc set-ups in actual chemical plants, as well as some close-up views showing details of bursting discs and holders.

will constitute a new field for private investment.

**Sulphuric and hydrochloric acid** are needed for the treatment of tin ores and consideration is being given to the establishment of plants for their manufacture. It is also desirable to establish one or more plants for the manufacture of calcium carbide, which is used in the mines and for which raw materials area available domestically.

**Leather.** There are numerous tanneries throughout the country. One has been installed in Cochabamba by a group of Hungarian immigrants and produces goods of high quality, some of which are exported. There is an urgent need to establish a tannin manufacturing plant to supply the domestic market and for export. Raw material is abundant and the Government wishes to give all possible assistance.

**Cement.** The existing plant is antiquated, but the new one now being installed in Sucre will probably be finished in 1956. The existing consumption does not exceed 20 lb. *per capita* p.a.; this is due to inadequate production rather than lack of demand and the figure could be raised to at least 55 lb. The Bolivian Development Corporation has initiated conversations with the French banking firm of Lambert, which appears to be interested in the installation of a new factory in Cochabamba. By utilising the present output of asbestos, factories for the production of fibre-cement pipes, sheeting, roofing tiles, etc., can be installed.

# Chemical Engineering Possibilities of CHROMATOGRAPHY in Industry

By T. I. Williams, M.A., B.Sc., D.Phil., F.R.I.C.

*The time may not be far away when chromatographic procedures, hitherto used mainly as a laboratory tool, will be called for in certain industrial separations. Dr. Williams, a leading authority on chromatography, points out some of the directions in which these techniques may be applied on the larger scale. He touches on some of the practical problems and economic considerations.*

THE chemical industry generally is not slow to avail itself of useful new techniques and it is therefore not surprising to find that it now very extensively employs chromatography—in its various modern forms—as a method of analysis. Apart from its great value in fundamental research, chromatography is an invaluable aid to industrial chemistry because of its extreme simplicity and the readiness with which it lends itself to the repetitive analysis of similar mixtures, which is so important in process control.

More surprising, in view of the method's extraordinary record of successes as a preparative method in the laboratory, is the fact that it has hitherto found surprisingly little application as a large-scale manufacturing process, a use for which it seems, at first sight, to be very suitable. The industrial chemist's life is, after all, very largely concerned with just the kind of problems with which chromatography seems most fitted to deal—the removal and recovery of impurities and the separation of the various components of mixtures.

This lack of interest is, however, rather more apparent than real. Although very little has been published, there has been much serious consideration of the matter in chemical engineering circles in many countries and to some extent it is inherent difficulties, rather than lack of enthusiasm, which have hitherto retarded progress. These difficulties come for the most part under one of two headings. On the one hand, there still seems to be only a very limited range of industrial separations where chromatography seems to offer a notable advantage over other processes already established and known to be reliable and satisfactory. Clearly this offers little incentive for the investigation of novel and quite untried processes. On the other hand, chromatography is still essentially a batch process, while the chemical engineer's

natural preference is for continuous processes.

Nevertheless, it is undoubtedly timely to consider the industrial possibilities of chromatography, for neither of the factors mentioned above is immutable. Chemical technology is advancing so rapidly today that industrial separations for which only chromatography is appropriate may arise at any moment. A case in point is perhaps already to be found in the metallurgy of zirconium, which has suddenly become needed in very considerable quantities in the field of nuclear power. As ordinarily prepared, zirconium contains some 2% of hafnium, which must be eliminated for atomic energy work because of its relatively high absorption cross-section for thermal neutrons. The extraordinary similarity of the two metals makes their separation by conventional methods very difficult, and ion-exchange chromatography seems to be a very likely answer for industrial requirements.

Again, although chromatography as now carried out is certainly a batch process, there seem to be no grounds for dogmatic assertions, sometimes voiced, that this is an inherent quality of the method and that it is pointless to seek to obviate it. On the contrary, various promising ideas—based generally upon counter-current devices—have recently been put forward to make the process a continuously operated one. Moreover, it is not difficult to imagine circumstances in which a batch process would be tolerable. Recent technological developments provide numerous examples—of which zirconium has already been cited—of a sudden urgent demand for a substance not readily obtainable. In many such instances the cost of the extraction process may be, if not immaterial, at least relatively unimportant compared with its effectiveness and ready availability. Moreover, batch processes have an already

established place, notably in the preparation of many fine chemicals for which the demand may be slight. In such instances there is no question of supply keeping strictly in step with demand; a quantity of material is made and the operation is not repeated until the first batch is nearing exhaustion.

## Early connections of chromatography with industrial practice

It is perhaps worth while to recall that, in seeking to apply chromatography as a preparative process in chemical technology, we are not attempting something novel, but retracing steps first taken as much as 70 years ago. One can trace a direct connection between early industrial practice and the adsorption methods of chromatographic analysis which first found extensive use in the 1930s. In 1886, C. Engler and M. Boehm described how petroleum jelly could be manufactured by passing crude petroleum through columns of charcoal. They observed that unsaturated hydrocarbons were retained—at levels corresponding to their double-bondedness—on the column, while the effluent consisted largely of saturated substances. Ten years later, D. T. Day, pondering the problem of why natural petroleum varies so considerably in its composition according to its source, concluded that the explanation was to be found in the occurrence on a gigantic scale in nature of processes such as Engler and Boehm had introduced into the petroleum industry. Day was quick to see the analytical possibilities of the process, describing it fully in 1900, and rather paradoxically it is these, rather than the industrial applications that inspired them, which became generally known and, ultimately, so very widely used.

It may be remarked, too, that another form of chromatography which has now become exceedingly important in analysis—paper partition chromatography—was also inspired by indus-

trial practice. The original work of F. F. Runge, published around 1855, had its source in the custom of workers in the dyeing trade to test the quality of dye baths by allowing a spot of the liquid to fall upon a piece of absorbent cloth or paper; the form of the concentric rings which appeared as the solution was drawn outwards by capillary action gave an indication of the quality of the dye.

### Industrial separations achieved

Setting aside for the moment the question of both economics and continuity of operation, there is ample evidence that the carrying out of chromatographic separations by conventional apparatus on a considerable scale presents no insuperable difficulties. Thus streptomycin has been purified industrially by chromatography on large columns of alumina, using methyl alcohol as the solvent. The principal plant pigments—chlorophyll, xanthophyll and carotene—have been effectively separated upon large columns—each about  $12 \times 3$  ft.—of carbon black, using hexane as the initial solvent and using a hexane-isopropanol mixture in the later stages of development. Chromatography has been used also for the preparation of amino acids for research purposes. This seems to be an almost ideal use for the conventional column. Here the products are sufficiently valuable for the cost of the extraction to be relatively unimportant and the quantities required are not so large that serious practical difficulties are encountered by magnification of ordinary laboratory apparatus. The original separation of vitamin K was carried out almost wholly by chromatographic methods, some of them entailing the use of columns containing 400 lb. of adsorbent. In the United States, 50-ft. columns have been used to separate petroleum fractions. During the last war, in the plutonium project, batteries of 9-ft. columns were used for the separation of some rare earths.

### Scaling-up difficulties

Nevertheless, experience with large chromatographic columns is sufficient to show that their size cannot be increased indefinitely without creating difficulties. Channelling, which can be troublesome with even quite small columns if they are not very carefully packed, quickly becomes serious and leads to distortion of the zones. The relatively prolonged development necessary to form a liquid chromatogram—the most satisfactory method of collecting the various fractions from

a large column—aggravates this distortion and leads to overlapping of the zones and collection of mixed fractions. Various devices have been proposed to improve the uniformity of packing—such as continuous agitation as the adsorbent is added—but the problem is clearly not an easy one to solve. One interesting proposal entails the use of a cylinder of adsorbent instead of a column, the solution flowing radially outwards from a central channel under the compulsion of centrifugal force. The use of this system is claimed to give not only a more even packing and accelerated flow but a greater throughput per unit of adsorbent and greater compactness. The zones are, of course, formed in this case as a series of hollow concentric cylinders. The full patent on this device provided for the adsorbent to be divided up among a series of quickly exchanged annular containers having perforated walls to allow free flow of liquid.

Accelerated flow may, of course, be obtained directly by increased pressure and this is relatively easily obtainable with columns of almost any size, in contrast with the centrifugal method which, for obvious engineering reasons, presents rapidly increasing mechanical problems as the weight of apparatus to be rotated is increased. In general it seems that with adsorption columns equilibrium is normally attained so quickly that within wide limits increased rate of flow does not affect the nature of the chromatogram unless, of course, it sets up local heating or other disturbing effects. With ion-exchange columns, however, this may not be true, especially in the case of some of the larger organic molecules, and the rate of attainment of equilibrium may be a major factor determining the speed of the process as a whole.

### Batch and continuous processes

It seems evident that, although a continuous process is desirable, and possibly essential, for any application of chromatography to industrial processes handling large quantities of raw materials—for example, the refining of petroleum fractions—there is room in the fine chemical field for batchwise chromatography in the preparation of such materials as vitamins, hormones and antibiotics when the price is high and the weight of material involved relatively low. Yet, even here, economic factors which may be relatively unimportant on the laboratory scale assume a considerable importance—a fact which tends to be forgotten

by some of the warmer advocates of industrial chromatography. For any but the smallest concerns, capital outlay on adsorbents—generally alumina, but often cheaper substances can be used—need not be a serious matter, as this can serve a wide variety of chromatographic and other uses and it is easily regenerated. Outlay on solvents is, however, a more serious matter. Relatively large volumes will be required, since the use of very dilute solutions, say 1 to 5%, is the general practice. Organic solvents are costly, some loss is inevitable in each cycle of operations and if the work is discontinued it may not always be easy to dispose advantageously of the surplus. Processes involving aqueous solutions are therefore particularly interesting. Possibly, therefore, reversed-phase partition chromatography, which has been relatively little explored but seems capable of very diverse applications, would reward study. Here the bulk of the solvent is water and only a small volume of organic solvent, firmly locked up in the material of the column, is required. For the same reason ion-exchange chromatography, for example, of some of the rarer metals required in small quantities for special purposes, seems technologically worth investigation. Again, gas-phase chromatography, in which volatile materials are diluted with nitrogen gas, may prove to be a useful preparative method for relatively small quantities of material.

In a recent article on gas-liquid chromatography (CHEMICAL & PROCESS ENGINEERING, 1955, 36 (3), 95), A. T. James has discussed the possibility of scaling-up the method to deal with considerable quantities. Although insufficient study has been made to determine the point at which apparatus of the present design becomes unduly cumbersome, there should be no difficulty in making columns capable of dealing with at least 100 g. of one substance. While such a quantity is very small in absolute terms it could, in terms, for example, of physiological activity or radioactivity, be very significant. Thus, 100 g. of vitamin C is a man's average requirement for about ten years. As the method is capable of application to all substances which can be distilled, it clearly has very wide scope.

The development of continuous chromatographic processes does not seem necessarily impossible. For example, in a method of foam chromatography—in which gas is bubbled continuously upward through a column—zones form at fixed levels in the foam and may be continuously withdrawn.



Similar counter-current methods using powdered adsorbents might also be used.

Various processes which are capable of continuous operation, although not continuous in the chemical engineering sense of the term, have been devised. These involve the use of several adsorption columns, some being processed and some standing idle as the remainder are in active use for the formation of chromatograms. The difficulty here is, of course, that only a fraction of the adsorbent, depending upon the proportion of columns in active use at any one time, is actually earning its keep. Where relatively expensive adsorbents or ion-exchange materials are being used the resulting cost may be prohibitive.

### Conclusion

The disappointing absence of large-scale applications of chromatography after it has so strikingly demonstrated

its preparative capabilities in so many different fields evidently springs neither from the indifference of the chemical engineer nor from any shortcoming in the method itself. That it is at present essentially a batch process seems neither incapable of being overcome nor an absolute bar to large-scale application. Lack of progress seems rather to derive from lack of industrial chemical problems which cannot be solved by conventional and well-tried methods. To apply chromatography in industry for preparative work is still decidedly a step into the unknown and consequently one not eagerly taken. But the pace of technological development is such that at any moment such a problem might arise—indeed, the separation of hafnium and zirconium which has been discussed may already furnish an example—and it is certainly not too early to be thinking of large-scale applications. There

seem to be, as has been shown, no inherent insuperable difficulties, but little progress seems to have been made on the design side. Ideas generally seem still to rise little above a great magnification of laboratory apparatus, but clearly an industrial chromatographic separator need bear no more resemblance to its laboratory counterpart than an industrial still does to a round-bottomed flask and Liebig condenser. What seems to be needed is for chemical engineers, especially those with experience in the adsorbent field, to turn their attention from the principles of large-scale chromatography to the design of apparatus for realising it. There seems every likelihood that when the time is ripe chromatography will establish a place in chemical technology, and it is desirable that when the need for it arises some ideas as to methods of procedure should already exist.

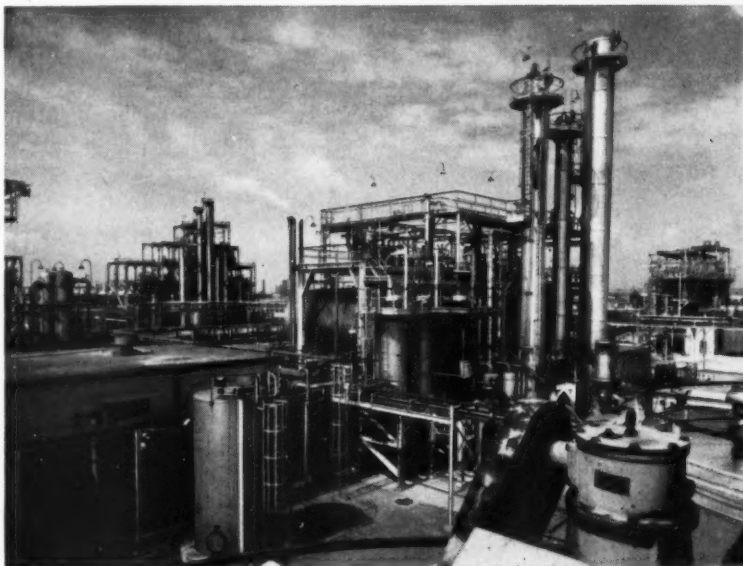
## PETRO-CHEMICALS EXPANSION

### British Petroleum Chemicals' New Projects at Grangemouth

**P**LANs for a major expansion of alcohol and petroleum chemicals production at Grangemouth (Scotland) were announced by British Petroleum Chemicals Ltd. recently. New plants, for which orders have been placed, will not only double the make of olefines but will also add to the list of petroleum chemicals already being made by the company.

An important part of the new plans is a new plant for extracting butadiene as a raw material for synthetic rubber and other plastics production. This seems significant in view of Monsanto Chemicals' intention to undertake the manufacture of synthetic rubber. Monomeric styrene has been produced at Grangemouth by Forth Chemicals Ltd.—jointly owned by B.P.C. and Monsanto Chemicals Ltd.—since 1953 and is used as a raw material for polystyrene production. Plans, previously announced, to more than double the capacity of the styrene plant are now well on the way to fulfilment.

The expansion plans also affect a plant, now well under construction, for the manufacture of an alkylate (dodecylbenzene) as an intermediate for synthetic detergent manufacture. This plant will be operated by Grange Chemicals Ltd., the company recently formed by British Petroleum Chemicals Ltd. and Oronite Chemical Co.,



General view of the British Petroleum Chemicals' plant at Grangemouth, showing the No. 1 ethanol plant (centre), the No. 1 cracking unit (left background) and the isopropyl alcohol unit on the extreme right.

U.S.A. The plant will convert tetrapropylene and benzene into dodecylbenzene by a catalytic process.

### Background to expansion plans

It was in 1951 that British Petroleum Chemicals commenced its operations

in a plant adjoining the newly expanded Grangemouth refinery of the British Petroleum Co. Ltd. The primary process is the cracking of a petroleum fraction, the main products being two reactive hydrocarbon gases, ethylene and propylene. British



These two photographs of the Grangemouth plant show (left) the interior of the compressor house of the No. 1 cracking unit and (right) one of the main control rooms.

Petroleum Chemicals produces synthetic ethyl alcohol from the ethylene, and converts the propylene to isopropyl alcohol (used as an intermediate for acetone and general chemical manufacture). Other quantities of ethylene are piped to the adjoining styrene monomer plant of Forth Chemicals.

In addition to ethylene and propylene, the mixture of hydrocarbons produced in the cracker contains a number of other potentially valuable compounds. One of these is polymerised in a small plant to make dicyclopentadiene, which is sold for use particularly in the preparation of synthetic drying oils for the paint industry; another important use is for insecticides. Until now, the remaining compounds have not been extracted.

### The new projects

The expansion, together with the necessary off-site facilities, is expected to cost over £8 million. The plans are aimed at:

- ★ Increased production of **ethylene and propylene** (and corresponding increase in the amounts of by-product hydrocarbons) by provision of a second cracking unit and a second gas-separation plant. The amount of ethylene available will satisfy the needs of the various Grangemouth plants and leave some surplus for sales to other users.
- ★ Extraction of **butadiene**, by the provision of a new unit to treat a C<sub>4</sub> stream drawn from the two gas-separation plants.
- ★ Increased **ethyl alcohol** production, by duplication of the existing plant.

★ Polymerisation of part of the propylene to **tetrapropylene** (an intermediate used in the production of detergent alkylate). The new tetramer plant will satisfy the requirements of the detergent alkylate plant and will provide, in addition, a surplus for sale.

The original cracking and gas-separation plants and the synthetic alcohol plant have all operated very successfully and at above design capacity so that, apart from minor improvements resulting from practical experience, it has been decided to build new units which are essentially duplicates of the existing ones.

### Raw materials

For the British Petroleum Chemicals' plants, increased amounts of light petroleum fractions will be supplied from Grangemouth petroleum refinery. For the Grange Chemicals' plant,

tetrapropylene will be forthcoming from the new tetramer plant and also from the petroleum refinery.

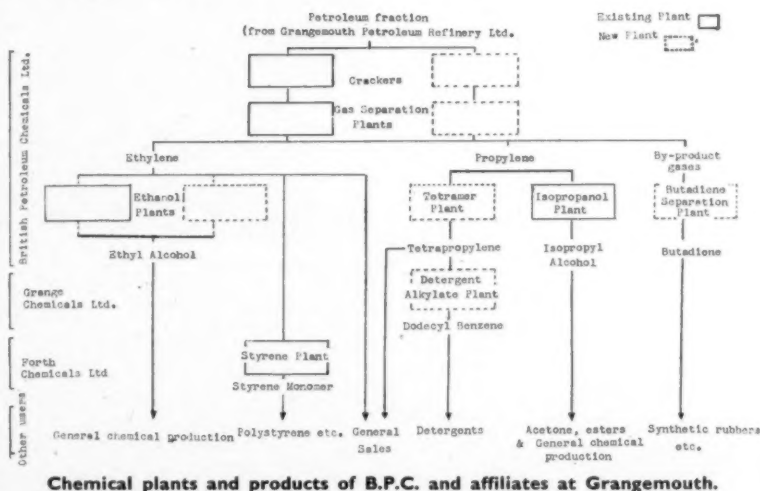
Benzene is available from indigenous sources.

### Construction contracts

The original (1951) plant of British Petroleum Chemicals was designed and constructed by Stone & Webster Engineering Corporation and its U.K. affiliates, E. B. Badger & Sons Ltd. A contract has already been placed with Stone & Webster for the design and construction of the extensions, with the exception of the butadiene separation plant, which will be built and engineered by the Kellogg International Corporation.

These plants are scheduled for completion between June 1956 and June 1957.

The Grange Chemicals' plant is also  
(Concluded on page 302)



# FRACTIONAL DISTILLATION

*Calculations; bubble caps; packed columns; perforated plates; batch distillation; extractive and azeotropic distillation; laboratory applications, etc.*

By H. H. M. Jones, B.Sc., A.M.I.CHEM.E.

IT is again convenient to classify the various aspects of this unit process, and new papers, etc., can then be reviewed under each section. In some instances it is advisable to refer to earlier work to draw the necessary comparisons.

## Calculations

The rational analysis of the unit process was carried forward by Gaumann *et al.*<sup>1</sup> who developed a method for resolving multi-component distillation by means of matrices, these being especially suitable for machine computation. Computers were also suggested by Opler and Heitz<sup>2</sup> for simplifying plate-to-plate calculations. Orlicek and Voetter<sup>3</sup> have a method half mathematical, half graphical and relatively simple. In addition, in this instance, differing latent heats, temperature and pressure gradients and heat losses can be taken into account. A different approach is that of Riediger,<sup>4</sup> who, using data already published,<sup>5</sup> calculates a fractionating column with respect to equilibrium concentrations, volatility coefficients, boiling and dew points.

Quick methods of calculation are proposed by Mayer,<sup>6</sup> who uses a graphical slide-rule method which can also be adapted to shorter plate-to-plate calculations where these are necessary for reasons of accuracy; and Eduljee,<sup>7</sup> who combines approximate methods from several sources into a recommended design procedure. An important aspect of multi-component separation, the interpretation of minimum reflux ratio conditions, is discussed by Franklin and Forsyth,<sup>8</sup> taking as a basis the Underwood equation.

Voetter and Orlicek<sup>9</sup> express efficiency in terms of a 'transmission unit' and apply this general principle to ternary mixtures. These are also considered by Schotte and Selke,<sup>10</sup> who use a method similar to Cope and Lewis.<sup>11</sup> On a nomograph for the equilibrium relations, operating lines are superimposed. Procedures for

various solutions are given depending on the key components.

A graphical interpretation of ternary separation is given by Lee and Kammermeyer<sup>12</sup> which is applicable to ideal and non-ideal mixtures. The procedure is simplified and its application to azeotropic and extractive distillation is discussed. An important application of ternary fractionation is the separation of liquid air, and Clare *et al.*<sup>13</sup> review existing design methods and propose a new one. It is based on a three-component enthalpy composition model resembling a unilateral prism for the nitrogen-argon-oxygen system. Examples of its application are given.

In the field of binary fractionation, Teller<sup>14</sup> offers a comprehensive review of the subject including adaptation to plant problems. Stage and Juilfs<sup>15</sup> deal with the optimum characteristics of binary mixtures. The application of temperature pressure relationships, Clausius-Clapeyron equation and Truton's rule to 130 hydrocarbons is given. The same authors<sup>16</sup> present an accurate graphical method for the evaluation of plate-type columns involving the application of equilibrium curves for the calculation of plates for any ethyl alcohol and water mixture at infinite reflux. Santos<sup>17</sup> under similar conditions compares the Dodge and Fenske equations with each other and the McCabe-Thiele method for plate calculation. Agreement is only good with ideal mixtures.

In another review of the calculation of the optimum characteristic of fractionation columns by Stage and Juilfs<sup>18</sup> the emphasis is on kinetic as well as thermodynamic relationships. It is confined to laboratory columns, and the estimation of the number of separation units for differing types of packing.

Watson<sup>19</sup> suggests a modification of the McCabe-Thiele diagram where  $y - x$  is plotted against  $x$  for greater precision and  $z$  versus  $x$  from the equation  $0z = x_d - y$  on the same diagram;  $z$  is the theoretical vapour enrichment per plate.

## Bubble-cap plates

The emergence of new types of contacting devices for gases and liquids has not lessened interest in the most widely used one. More aspects of its function have been investigated.

The mechanism of mass transfer is investigated by West *et al.*<sup>20</sup> Values of  $k_{ga}$  by the humidification of air with water and of  $k_{la}$  by the absorption of  $\text{CO}_2$  in water and desorption with air are given. Highie's equation<sup>21</sup> was used to calculate individual mass-transfer coefficients. Eventually, an equation was derived relating Murphree point efficiency to vapour and liquid diffusivities, fraction of voids in foam, liquid level, overall gas velocity and bubble radii. Agreement with experiment was poor, due probably to inaccuracy in void fraction and bubble radius. However, it was deduced that orifice size and shape seem relatively unimportant in fixing bubble size on a well-agitated plate. On the related question of slot opening and flow rate, Dee<sup>22</sup> uses data from a previous paper<sup>23</sup> for  $\frac{3}{16}$ -in. slots in acetic acid water solutions to show that they are in agreement with those for air through 2-in. slots in water reported by Cross and Ryder.<sup>24</sup>

The pressure drop through bubble-cap plates is the subject of a study by Garner *et al.*<sup>25</sup> who show that it is greater than the height of liquid above the slots. It is postulated that the extra pressure drop is due to the formation of interfacial surface of the bubbles. Cylindrical bubbles show a reduction of pressure as compared with the equivalent spherical formation. A more limited investigation was reported by Canley *et al.*<sup>26</sup> where pressure drop was determined separately as a function of weir height and as skirt clearance. Data only applicable to the 2-ft.-diameter plate were used.

Blaukut<sup>27</sup> develops a formula and applies it to a 48-plate column; from it the pressure difference can be predetermined by controlling the vacuum. The head pressure varied from 100 to



4,000 kg./sq.m. in the separation of phenol and *o*-cresol with relatively small change in pot temperature (129 to 177°C.).

Design methods for bubble-cap plates are provided by Omori,<sup>28</sup> who emphasises fluid mechanics, and by Atkin<sup>29</sup> with a basis of observation of commercial units with a wide range of operating conditions. An important factor in practical design is plate efficiency, called exchange efficiency by Hansen,<sup>30</sup> who presents a graphical means of assessing it. The same value is obtained whether the vapour or liquid enrichment is considered.

The efficiency of *Kaskade* plates is compared with bubble-cap trays by Garner *et al.*<sup>31</sup> They find that only at low loading is the conventional design superior. The latter, however, has a maximum efficiency at about 1 ft./sec. overall vapour velocity. The newer design does not show this limitation and, in the higher range of vapour flow for the same conditions of efficiency and pressure drop, it has the higher capacity.

#### Packed columns

An extensive and practical review has been prepared by Zenz,<sup>32</sup> who presents the various design methods and works out an example. A literature survey of the subject was carried out by Smith.<sup>33</sup>

The fundamentals of the function of packed columns were studied by García and Barreiro,<sup>34</sup> who analyse experimental data for the separation of benzene and carbon tetrachloride with the aid of an equation developed by Simon and Govinda Rau.<sup>35</sup> Mass transfer is shown to be controlled by the liquid film. The same conclusion, on more general grounds, was arrived at by Stuke.<sup>36</sup>

The hydrodynamics of gas and liquid flow through packed columns in both rectification and gas absorption were studied by Kafarov and Blyakhan.<sup>37</sup> The pressure drop through irrigated columns was correlated to that for non-irrigated (previously determined) by means of a factor which was a function of reflux ratio, vapour and liquid densities and viscosities. The limiting capacity for rectification was less than for absorption. This last factor was also examined by Fujita *et al.*,<sup>38</sup> who included also liquid/liquid flow. The gas was always treated as the continuous phase. The correlation contains the usual factors: gas and liquid viscosities, overall velocities, surface area, voids, etc. The last characteristic of a packing is examined theoretically and practically

by Hirai;<sup>39</sup> two equations are derived, one for spherical and the other for irregular pieces.

The H.E.T.P. of a packing is a characteristic usually so much dependent on conditions of operation that the equation enabling its prediction (Murch<sup>40</sup>) is valuable. It is based on the data from 148 experiments and it is claimed that for 95% of cases the H.E.T.P. can be calculated from it to within  $\pm 10\%$ .

An indication of the influence of the composition of the mixture on the H.E.T.P. is shown by the experiments of Chahvekilian.<sup>41</sup> Here it increased from 25 cm. to over 50 cm. as the feed concentration of carbon tetrachloride in a mixture with benzene varied from 10 to 80%. These results are explained by inconsistency of  $k_{ga}$  and  $k_{la}$  by Chilton.<sup>42</sup>

The H.E.T.P. values for metal helices and ceramic saddles and rings in laboratory columns were studied by Walsh *et al.*<sup>43</sup> The columns were all preflooded because without this precaution very varying results were obtained. The efficiency of the columns was found to be independent of pressure over a range 200 to 760 mm. H.E.T.P. for helices varied from 2 to 3.6 in., saddles 4.2 to 5.2 in. and rings 5.2 to 8.2 in.

The Stedman packing and its application to a laboratory column is described in detail by Nakanishi.<sup>44</sup> A more practical paper is by Bragg,<sup>45</sup> who gives an equation correlating the number of theoretical plates per foot with the rate of reflux. The packing size ranges from  $\frac{3}{8}$  in. conical to 11 ft. hexagonal pyramid packing. Within the limits of the suitable liquid loading of a column the number of plates is independent of column diameter.

An effect little investigated is that of the packing supports. Leva *et al.*<sup>46</sup> showed that the support-free area can vary from 23 to 92% and that the design of the support can influence pressure drop by as much as 50%, especially if irrigation rates are high.

The hold-up in a packed tower was analysed by Otake and Kimiura<sup>47</sup> from previous experimental data. The correlation consists of gas/liquid velocities against a set of dimensionless groups obtained analytically.

The low pressure drop in packed columns is of advantage in vacuum distillation and Peters and Cannon<sup>48</sup> give H.E.T.P. values for various  $\frac{1}{4}$ -in. packings between 10 and 735 mm. absolute. Feldman *et al.*<sup>49</sup> compare a *Heli-Pak* No. 2917 column with other packings.

Finally, the rectification of various

mixtures at atmospheric and sub-atmospheric pressure was investigated by Kirschbaum and David.<sup>50</sup> H.E.T.P. and pressure drop were plotted against vapour velocity.

#### Perforated plates

Performance data and design notes based on perforated-plate columns in service are given by Lee.<sup>51, 52</sup> He claims that effective design can widen the application of this type of contacting device which, in many cases, is cheaper and more effective than the bubble cap.

The relation between hold-up and pressure drop for a perforated plate is the subject of an investigation by Kamei *et al.*<sup>53</sup> An experimental equation based on the sum of the dry plate pressure drop and that due to the foaming layer is presented. Kamei, in association with other workers,<sup>54</sup> examines the critical feature of this type of plate, namely the minimum allowable vapour velocity. The effect of hole diameter, pitch, liquid overflow rate and slope of the plate are investigated on a plate 59 cm. in diameter. Formulae correlating the variables are given. It is found that slope has less effect on uniform distribution than was previously assumed.

Three entirely new tray designs are suggested by Bowles,<sup>55</sup> Nutter<sup>56</sup> and Thrift.<sup>57</sup> These are all variations of the bubble-cap tray, but use lifting on pivotal values to provide flexibility in design and operation.

#### Batch distillation

The comparison between batch and continuous distillation is made by Ellis and Shelton in two papers.<sup>58, 59</sup> A mixture of 80% phenol and 20% *o*-cresol and a 99.5% phenol product is assumed. The yields at various reflux ratios (the average is taken for batch distillation) are compared for 35, 50 and 70 plates. As plate number nears a minimum the batch method requires less plates; at reflux ratios approaching the minimum that of the continuous method has the lower value. At low hold-up the advantage of continuous working diminishes.

The second paper describes experiences with columns of small numbers of plates and a 90% yield of the product. It is decided that the relative advantage of either method depends on the yield and the number of theoretical plates in each column.

The advantages of both methods are combined by Kiguchi,<sup>60</sup> whose apparatus operates over a wide range of capacity, pressure and temperature.

General reviews of batch distil-

lation are presented by Omori,<sup>61</sup> whose treatment is theoretical, and Kwang-Chu,<sup>62</sup> who shows a short-cut method for calculating the batch distillation of complex mixtures.

The same problem is tackled by Ellis,<sup>63</sup> who extends Gilliland's correlation<sup>64</sup> to batch distillation. This method is useful for cases of close-boiling components and can also be used for feed-plate location in continuous distillation.

The influence of various factors on the sharpness of separation was investigated by Zuiderweg.<sup>65</sup> Hold-up affects it favourably at high reflux ratios and *vice versa*, but at very high hold-up (equivalent to 50% of the charge) reflux ratio was without effect. The experimental data were correlated by expressing 'pole height' as varying with hold-up, volatility, fraction of residue at 50% mol. fraction in product and a fractionating factor

$$[E = \alpha(N + 1)].$$

When the product composition is kept constant, another approach, as exemplified by Hibino,<sup>66</sup> is possible. Here an optimum number of plates can be deduced from a graphical representation of time for distillation against number of plates at varying relative volatility. The family of curves is hyperbolic and asymptotic to minimum values of the variables.

The effect of vapour velocity on plate number (other factors being held constant) is the subject of a paper by García and Barreiro.<sup>67</sup> Their results with a carbon tetrachloride and benzene mixture were similar to those found by Kirschbaum<sup>68</sup> with ethyl alcohol and water.

### Extractive and azeotropic distillation

In two papers, Dummett<sup>69</sup> reviews the basis of extractive and azeotropic distillation and goes on to describe industrial applications. A detailed exposition of the mechanics of this branch of fractionation with references is by Sanghvi and Tayabkhan.<sup>70</sup> Paris<sup>71</sup> lays down criteria for the choice, firstly, of a separation process (liquid/liquid extraction, azeotropic or extractive distillation), and secondly, of the best solvent. Emphasis is on the practical aspect of heat economy throughout the process. The application of extractive distillation of acetic acid and water, but using sulphuric acid instead of ethyl acetate, is presented by Teshima *et al.*<sup>72</sup> It seems hardly of practical significance, because of the high optimum concentration of the solvent.



[Q.V.F. Ltd.]

The photograph shows a Q.V.F. 12-in. reflux distillation head of the swinging-bucket type, with solenoid and reflux ratio timer. The timer (manufactured by Elcontrol Ltd.) is designed to energise the actuating solenoid cyclically, enabling reflux ratios, both multiple and fractional, to be applied. These range from 50:1 to .02:1. The combination is claimed by the makers to be extremely versatile.

Gel'perin and Novikova<sup>73</sup> extend the method of stepwise rectification at two different pressures for the separation of binary azeotropes. The method is applied to maximum and minimum azeotropes and experimental results for both cases are given.

Gándara<sup>74</sup> gives a method for graphical calculation of the separation of a binary solution by means of an entrainer. The latter may be supplied with the reflux alone or also with the feed. There is the limitation that it is assumed immiscible with the top product.

The practical aspect is provided by Carlson *et al.*,<sup>75</sup> who recommend the use of water as a solvent for separating mixtures of compounds resulting from, say, the oxo reaction into classes of compounds, *e.g.* alcohols from ketones. In further separation within a class the compound of higher molecular weight is rendered more volatile by similar methods.

### Laboratory applications

How to set up a pilot-scale distillation laboratory is described by Friden and Stack.<sup>76</sup> All the necessary apparatus, including instrumentation, is specified. Schultze and Stage<sup>77</sup> give a more detailed specification for a general-purpose laboratory column.

The principles of laboratory fractionation are presented by Jaulines,<sup>78</sup> while Hetherington<sup>79</sup> discusses the design of the four parts of an apparatus (column, reboiler, condenser and reflux head) and then illustrates this by details of the azeotropic separation of some tar bases.

The determination of accurate vapour/liquid equilibrium data for homogeneous mixtures continues to occupy attention. Ellis extends the application of a device already reported.<sup>80</sup> The still, which eliminated several sources of error and provided thermodynamically consistent data at atmospheric pressure, has now been modified to provide results down to 100 mm. absolute total pressure. Its use is described by Ellis and Froome.<sup>81</sup> In a later paper, Ellis has again put forward an improved type of equilibrium still<sup>82</sup> and its advantages and disadvantages are compared with those devised by Othmer and Gillespie.

An apparatus that can also be used for heterogeneous mixtures is described by Koller.<sup>83</sup> The emphasis is on improved experimental method which does not require that the binary mixture be boiling.

Another still involving the use of two separate evaporation chambers is due to Bukala and Majewski.<sup>84</sup> They compare its accuracy with more conventional types.

There are two interesting papers relating to the insulation of laboratory columns. Junge<sup>85</sup> shows that the use of an evacuated jacket is not entirely effective because of air being absorbed by the glass. This requires heating above 400°C. during evacuation. He concludes that glass fibre is more effective and cheaper. Williams<sup>86</sup> suggests a jacket filled with a liquid boiling at a definite temperature under variable but controlled pressure. Great accuracy ( $\pm 0.25^\circ\text{C}.$ ) over wide temperature range is shown by an experimental example.

A special fractionating column for corrosive mixtures of liquefied gases is described by Orrick and Gibson.<sup>87</sup> This is of the Podbielniak type and constructed of nickel, bronze and Teflon.

Columns for operation at low pressure are compared by Jantzen and Wiekhorst<sup>88</sup> with special reference to the annular-slit type. They give the conditions necessary for separation and the design of a specific column.

A short-path fractionation unit suitable for continuous operation is reported by Utzinger.<sup>89</sup> The charge is recycled and exposed to distillation temperature for 10 sec. only.

Finally, an elegant control system for laboratory fractionation is described by Abegg.<sup>90</sup>

### Miscellaneous

A review covering the important trends in the past ten years or so in fractional distillation is presented by Dummett and Clifton.<sup>91, 92</sup>

Columns employing a rotary principle are still being used in laboratory investigation. Jost *et al.*<sup>93</sup> describe such a column with a rotor turning at the relatively low speed of 2,600 r.p.m., giving an H.E.T.P. of 1.20 cm. for a distilling rate of 690 cc./hr. Hold-up was 0.17 cc./theoretical plate. The column was equivalent to 43 theoretical plates and the pressure drop was less than 0.5 mm. A smaller column, distillation rate 20 to 60 cc./hr., was tested by Crozier *et al.*<sup>94</sup> They found that the number of theoretical plates fell sharply as the distillation rate was increased, but that this could partly be counteracted by increase of rotor speed. The column was 110 cm. long by 8 cm., equivalent to 30 to 40 plates. A vertical rotating apparatus consisting of alternate stationary and rotating cones placed end to end was suggested by Gel'perin and Khatsenko.<sup>95</sup> Output was low due to low linear speed of the cones.

Rollert<sup>96</sup> continues his study of a horizontal column.<sup>97</sup> Finite reflux can now be considered in the analytical procedure for calculation of the number of theoretical plates. On test runs the efficiency fell off sharply above the optimum reflux ratio.

The control of fractionation columns is treated empirically by Swietoslawski,<sup>98</sup> where temperatures at points on the Engler distillation curves of the product are used as criteria of column equilibrium. The use of intermittent product withdrawal on separation efficiency has been studied again; this time by Romanet.<sup>99</sup> Theoretically, there is a slight gain and a plot of amount of each withdrawal/hourly distillation rate against reflux ratio shows a point of maximum efficiency which is higher than that obtained by continuous withdrawal. Bauer and Orr<sup>100</sup> use the McCabe-Thiele diagram to determine the degree of control attainable in a binary distillation when feed composition is the variable.

General reviews of the recent advances in types of rectification apparatus are made by Freshwater,<sup>101</sup> who compares the newer *Kaskade* and *Benturi* with more conventional devices, and by Thormann.<sup>102</sup>

The *Turbogrid* tray is described<sup>103</sup> with comprehensive performance-test

data. Applications outside fractionation are also emphasised.

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## Ion Exchange

Ion exchange is a unit operation in which theory has outstripped practice. A book, written from the industrial viewpoint and showing the significance of recent equilibrium and kinetic studies to equipment and design, choice of exchanger and of operating conditions would be of very great value. This is not the kind of book which M. Eeckelaers, the technical director of a Belgian firm in the ion-exchange field, has written.\*

What he has provided is a sort of *vade mecum* for a young man who is just entering the ion-exchange business. The degree of ignorance which he has assumed in his reader, however, renders the book very largely irrelevant to a wider professional circle. Thus, to quote but a few examples, ionisation, pH and gramme-equivalent are each carefully defined, with examples. This treatment leaves little space for anything but a cursory examination of the history, theory and properties of ion exchange in the 42 pages given over to these topics. The discussion of equilibrium relationships, which closely follows Nachod's book, is perhaps adequate, although the effect of concentration on the distribution coefficient receives scant treatment, but the kinetics of the ion-exchange process goes virtually without mention. The section on properties of exchangers does not discuss their relative solubility and stability in highly reactive media—a feature of the greatest importance when many exchangers are very expensive. The casual printing of graphic formulae (e.g.  $\text{SO}_3\text{H}$  for  $\text{SO}_3\text{H}$ ) is most apparent in this section, but the printer can hardly be blamed for the reproduction of a formula for linear sulphonated polystyrene as a

representation of an exchanger cross-linked with divinyl benzene.

A chapter of 33 pages on 'Water' again contains many irrelevancies, but does give useful information on Continental water-treatment practice. It is unfortunate that no attempt is made at an economic comparison of methods. The preparation of potable water from brackish or sea water is discussed without reference to membrane electro-dialysis methods, although these are briefly mentioned elsewhere.

Two chapters (56 pages in all) follow on miscellaneous industrial applications, including the treatment of sugar, glycerol, milk, wine and fruit juices, the recovery of metals from solutions, and catalysis. The sections on sugar and glycerol collect together information on a number of techniques, with their advantages and disadvantages, and are a helpful contribution to the literature of the subject. The reviewer found this quite the most useful part of the book. The remaining topics are dealt with very briefly indeed.

A chapter on equipment (8 pages) is too scanty to be of much value, the section on acid-resisting pumps consisting essentially only of photographs of two well-known types. Two chapters (13 pages each) on specifications for regenerants and analytical methods are unnecessary in a book about ion exchange, as is the last chapter, which is a collection of tables containing, for example, a table of atomic weights and a comparison of thermometric scales.

The book concludes with a list of over 600 references, in the order quoted in the text, and an alphabetical index of authors quoted. It will be seen that, while it may be of service as an internal laboratory or factory handbook, it contains little material of interest to the outside professional

or general reader. By British standards, the price of nearly £2 for a paper-covered volume of less than 200 pages would be excessive.

J. M. HUTCHEON

## Chemical Behaviour of Aluminium

This volume\* may be considered as the second edition of a book which was published in 1935 in several languages by the Bureau International des Applications de l'Aluminium as the result of scientific and technical collaboration between the British, German, French and Swiss aluminium industries. The title of this first publication was 'The Use of Aluminium in the Chemical Industry and in the Manufacture of Food.' As 20 years have passed since the publication of this book, it has been obvious for some time that a need existed for a new book on the chemical behaviour of a metal which has gained in importance as a constructional material for the chemical engineer. This task has been undertaken by the Aluminium-Zentrale, Berlin, in collaboration with the groups belonging to the Centre International de Développement de l'Aluminium.

The book begins with a chapter on the corrosion of aluminium in general. It shows the influence of purity, heat treatment and surface conditions, and describes various types of corrosion, corrosion tests and methods of increasing the resistance to corrosion. The main part of the book is a detailed report on the behaviour of the various qualities of commercial aluminium in contact with all the substances that have ever been tested in this respect, including a great number of inorganic and organic substances, petroleum, essential oils, alkaloids, tanning extracts, sewage, fuel gases, waste gases, paints, synthetic resins, artificial fibres, anti-freeze mixtures, cosmetics and foodstuffs, to mention only some.

The book is valuable because it brings together a great deal of information that is widely dispersed in the scientific literature of several countries. An appendix of 53 pages presents in tabular form a summary of the behaviour of 99% aluminium and, where necessary, 99.8% aluminium and of aluminium protected by surface treatment or corrosion inhibitors in contact with over 1,000 different substances.

\**Das Chemische Verhalten von Aluminium* (the chemical behaviour of aluminium). Compiled by Aluminium-Zentrale e.V., Berlin. Aluminium-Verlag GmbH, Düsseldorf, 1955. Pp. 333 inc. index. DM 15.

# Some Chemical Engineering Problems of the IRON AND STEEL Industry

(Part 2)

By A. H. Leckie, PH.D., F.R.I.C., A.M.I.CHEM.E.

*Dr. Leckie discusses briefly some further instances of how chemical engineering can help to increase iron and steel productivity even more. He continues from where he left off in our June issue.*

IN spite of the long history of the open-hearth furnace, problems are still numerous. In recent years, much has been learnt about the aerodynamic design of the melting chamber<sup>14, 15, 16</sup> and burners,<sup>17, 18</sup> and the theory of heat transfer.<sup>19</sup> Modern furnaces are now invariably designed after careful consideration of the results of aerodynamic research (which was not always the case earlier), and it is now quite common practice to build a small-scale transparent 'Perspex' model of a proposed new furnace while it is being planned. By flow pattern experiments in the model, using cold water or air (plus a visualising medium such as aluminium particles or balsa dust) as a fluid medium, it is possible to determine aerodynamic faults in a design before the production furnace is built. Although aircraft and ship designers have used these techniques for many years, the correct similarity criteria for furnace designers have been sufficiently in doubt to delay their application to furnace problems until the problem received concentrated study after the war.

Whilst great advances have been made in what might be regarded as the 'physical engineering' of steel-making, less progress has been made in the chemical engineering problems of refining. It has already been mentioned that one of the difficulties the industry faces in the future is the need to make steel of still lower sulphur and phosphorus contents with raw materials which are likely to contain more of these elements. This leads to greater slag volumes and longer refining times in the open hearth which add seriously to the melting cost.

## Stirring problem in steel-making

There has been much fundamental work on the physical chemistry of the steel-making reactions, but less on the chemical engineering problems. It is generally assumed that stirring of the



[British Oxygen Co. Ltd.]

Fig. 5. Oxygen being blown on to the surface of the molten metal and slag in an open-hearth furnace. The oxygen pipe seen through the furnace door is water-cooled.

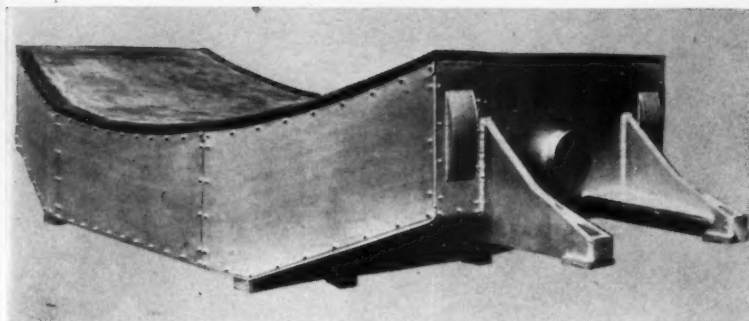
metal and slag would speed up refining, but it is difficult to see how any of the conventional mechanical stirring methods used in the chemical industry could be applied under furnace conditions. So far the only stirring effects attainable in practice are (i) the natural 'boil' of the carbon monoxide evolution as carbon is oxidised, and (ii) the stirring effect of oxygen injected under or on to the surface of the slag. Both these effects can take place over only part of the refining period. The injection of oxygen, now becoming a commonplace method of speeding up the removal of carbon, particularly to the low concentrations (under 0.1%) demanded for low-carbon sheet steel, brings problems both of fume emission and of refractory wear. Design of the jet to minimise splashing, and to give optimum penetration of the slag, is not yet finalised. In the U.S.A. some steel plants are finding it necessary to add elaborate waste gas cleaning equipment involving electrostatic precipitation to eliminate fumes in order to meet the requirements of the local authority, and this means great ex-

pense, and increases the cost of the steel substantially. Cheaper methods of removing the worst of the fume are needed.

One method of stirring which may have a future application to the open-hearth furnace is electromagnetic stirring by means of a low-frequency induction coil placed below the furnace bath. This method is already used successfully with electric arc furnaces<sup>20</sup> which, however, are much smaller than the average open-hearth furnace, but the problems of larger-scale operation should not be insuperable. Fig. 6 shows a coil unit used for this purpose beneath the hearth of an electric furnace.

## Cold metal process

Although discussion of steel-makers' problems here is mainly concerned with the hot metal process as used in integrated works because an increasing proportion of steel will be made in this way, and because more chemical engineering problems are involved, the cold metal process will continue in districts such as the industrial Mid-



[ASEA Electric Ltd.]

Fig. 6. A low-frequency stirring coil unit, curved so as to fit exactly below the hearth of an electric arc furnace.

lands, where large amounts of scrap arise from the local metal working industries. The cold metal process in future will tend to be mainly a scrap melting process with just enough cold pig iron brought in to give the necessary carbon boil and other qualities required in the steel. The chemical engineering problems of the cold metal process are less marked than these of the hot metal process because the charge contains more steel scrap and less pig iron, so the refining problems are less. Unless a fair amount of phosphoric pig iron is included in the charge (when phosphorus may become troublesome), the main problem is likely to be sulphur pick-up from the fuel, particularly when this is the high sulphur fuel oil which may have to be used to an increasing extent.

#### Desulphurisation of fuel

Since sulphur in the fuel used represents one of the main sources of sulphur in steel-making, increasing attention will have to be given to the possibility of desulphurising fuel. The problems of desulphurising fuel oil are more within the bounds of the oil industry than of the steel industry. Some account of them has been given recently.<sup>21</sup> Where coke oven gas is used as a furnace fuel the problem of desulphurising is one of conventional chemical engineering. Several works are either operating processes for desulphurising coke oven gas, or contemplating their installation. A description of the process used at Corby has been published recently.<sup>22</sup>

#### 'Duplexing'

Since the problem of slag bulk and refining time is becoming even more serious in the open-hearth furnace, it is not surprising that more and more attention is being paid to the possibility of carrying out some degree of pre-refining of iron between the blast

furnace and the steel furnace. The best known process of this kind is that widely known as 'duplexing,' but ladle refining is also coming to the fore.

The 'duplexing' process, well known in the U.S.A. and on the Continent, involves the partial blowing of the iron in a Bessemer converter before the metal is charged to the open-hearth steel furnace for final refining. Since in this process the metal charged to the open hearth may be well on the way to being finished steel, it is not surprising that very high output rates can be obtained from the latter unit.

Although a few works in Britain do 'duplex' from time to time, and a new plant has recently been installed at Consett,<sup>23</sup> the process has not become popular for general use. Most works consider the cost of operating what is in effect two steel-making units (both of which involve some loss of yield) in series are greater than the savings in melting cost in the open



Fig. 7. Pouring iron from a mixer into a transfer ladle for transport to the Bessemer converter. Sodium carbonate is often placed in this ladle prior to adding the metal in order to desulphurise the iron.

hearth. It is contended that it is better to operate Bessemer and open-hearth plants as separate units, and one advantage of this is that high-grade process scrap from the Bessemer plant can be returned as raw material for the open hearth. Again, much depends on local costs, and in future it is quite possible that price movements may favour some increase in 'duplexing.'

#### Mixers and ladle reactions

Whilst there are still plants where iron is taken straight from the blast furnace to the steel furnace, most integrated plants now use an intermediate vessel called a 'mixer' in which the iron coming from the blast furnace is stored until required for the open-hearth furnace or Bessemer converter. In fact, under most conditions a mixer is essential. Some of these mixers are 'inactive,' i.e. they are merely storage vessels in which little change in the composition of the iron occurs. Other mixers, known as 'active mixers,' are constructed on the lines of the open-hearth furnace itself, and the impurities in the iron ore reduced to a considerable degree both by oxidation and by dilution with scrap. Active mixers are generally used to remove some silicon from the iron, and attempts have been made, with limited success, to accelerate their refining action by the injection of oxygen or air into the molten metal.

However, the active mixer is quite costly to operate, and in any case neither type of mixer can deal to any great extent with the menace of increasing sulphur in the iron (except for such slight reduction as may take place through scrap dilution or the flotation of manganese sulphide). Interest has been growing in various means of partially purifying the iron in the transfer ladle by means of added reagents, or by means of an oxygen jet. Such processes do not remove the need for a mixer as a storage vessel, but they do permit the mixer to be of the cheaper 'inactive' type.

#### Reduction of silicon and phosphorus content

Substantial reductions of silicon can be made by blowing oxygen into the iron through a steel tube. The chief problem here is one of atmospheric pollution by the thick brown fumes of iron oxide which are produced. The oxide particle size in this fume is extremely fine, and only the application of elaborate and costly fume extraction and gas cleaning methods can deal with it effectively. Until a

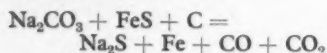


cheap way of reducing this fume trouble can be devised, this process, attractive as it is in many ways, is not likely to be practicable for works in densely populated areas, although it is already used in a few works in relatively isolated positions.

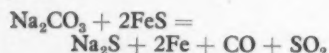
It may be that some reduction in the phosphorus content of high phosphorus irons may be possible by a similar technique, thus reducing the refining load on the open hearth. The free energy curves (derived from the ordinary thermodynamic formula  $\Delta G = H + T\Delta S$ ) for the oxidation of carbon and phosphorus,<sup>24</sup> suggest that at the temperature of molten iron phosphorus will not be removed to any appreciable extent until the carbon has been removed to a low concentration first, *i.e.* until the iron has already been converted into steel. However, the carbon-oxygen reaction appears under certain circumstances to be slow and, in fact, there have been numerous examples reported where appreciable dephosphorisation has occurred in the presence of carbon when injecting oxygen if a calcium ferrite slag is also present.<sup>25</sup> Whether or not preliminary dephosphorisation of iron will ever become a practicable process is still very much an open question.

### Sulphur removal

By far the greatest amount of research on ladle refining has been concentrated on sulphur removal. The only process which has so far proved successful as a routine operation for high tonnage production is the sodium carbonate process.<sup>26</sup> The reaction is generally assumed to be:



though from the strong smell of  $\text{SO}_2$  noticed during the operation, the reaction



may take place to some extent.

The operation is extremely simple since it is only necessary to place the required amount of soda in the ladle prior to tapping the furnace into it.

Chemically the process is very effective, it being easy to reduce the sulphur by 50%, but the chief difficulty in operating it is that of complete removal of the soda slag before the iron is taken on to the mixers and steel furnaces. There, the presence of traces of soda would do serious damage to the refractories.

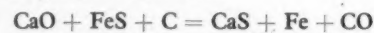
In present-day practice the slag is skimmed off with a rabble, which

means that either some residual slag is left on, or appreciable amounts of valuable iron are dragged off with the last traces of slag.

At present large-scale use of the soda process is limited to plants using the Bessemer steel-making process (where there is less risk of refractory damage) or to ironworks where the pig iron is cast into solid form for transport to the cold metal steel plants, as shown in Fig. 8. In casting the 'pigs,' residual soda slag is, of course, broken off the surface, or dissolved off in the water used for cooling the pigs.

Thus research is necessary to find an efficient means of separating the soda slag; for instance, it might be possible to discover some light porous refractory which could be scattered on the metal surface to mop up the traces of slag and then be raked off. Most of the known substances of this kind (*e.g.* vermiculite) melt at the temperatures involved. More elaborate ladles of the 'teapot' type have also been tried, but so far do not seem to have provided a solution.

Many other reagents for sulphur removal in the ladle have been suggested. A lime/alumina slag has been recommended,<sup>27</sup> together with the blowing in of an inert gas such as nitrogen, to give some stirring and to reduce the partial pressure of CO and so permit the reaction:



to approach completion. Agitation of the iron with solid lime in powder form has also been tried successfully on a pilot-plant scale.<sup>28</sup> Injection of calcium carbide has been another approach.<sup>29</sup> Metallic magnesium has also proved a successful desulphurising reagent for ladle use,<sup>30</sup> but here cost is the obstacle.

Whether any of these alternatives to the soda process will ever prove economic for large scale use is problematical. Clearly agitation with a liquid slag in an open ladle cannot be carried out for very long without the metal and slag freezing, and if an enclosed heated vessel is used costs rise steeply and one might as well send the iron straight on to the steel plant. The powdered lime process is regarded by many as having considerable promise, but since its success depends on the large specific surface of the powdered lime reagent, any blast furnace slag carried over from the blast furnaces with the iron is liable to 'slag' the lime and reduce its effectiveness.

The above very brief mention of some of the problems encountered in attempts to do some pre-refining to

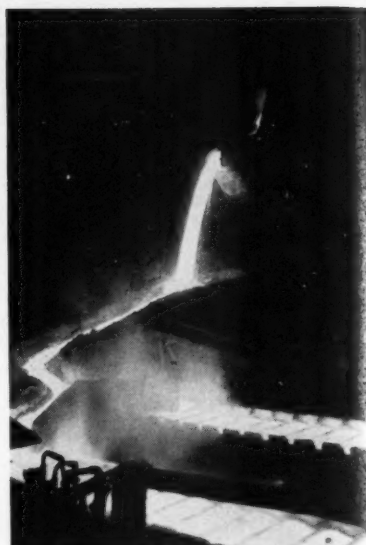


Fig. 8. A pig casting machine in action. The metal is poured into a series of moulds attached to a conveyor and cooled by water sprays.

reduce the metallurgical load imposed on the open-hearth furnace show how the chemical engineer interested in high temperature reactions can find numerous interesting problems in this field.

### Electric furnaces

Up to the present, the use of electric furnaces has been limited to the manufacture of the more specialised alloy steels which command a high price, and the chemistry of whose manufacture demands a furnace atmosphere control not governed by the high oxygen potential needed to burn a fuel efficiently. The value of these steels is such that the high cost of the electric power can be tolerated. The furnaces used in Britain are generally below 40 tons' capacity, though there is one 60-ton unit in the Sheffield area.

Much larger electric furnaces are used in the U.S.A. and American experience is beginning to suggest that by the use of these larger arc furnaces, which may approach the normal open hearth size of 100 to 200 tons, the consumption of power and electrodes can be reduced to a point where the arc furnace becomes competitive with the open hearth for a much wider range of steels.<sup>31, 32</sup> For instance, the ease with which sulphur can be removed in arc furnaces, owing to the low oxygen 'potential' in the furnace, suggests that this may be one solution to the problem of making low sulphur steel

from higher sulphur raw materials. The arc furnace provides some interesting problems in what might be called 'electro-chemical engineering,' since in certain circumstances the furnace and its contents of metal and slag, fed with conventional 3-phase a.c., may tend to act as a rectifier,<sup>33</sup> and some interesting electrical effects, perhaps linked with the composition of the steel and slag, can be observed.

If arc furnaces are to be used for a wider range of steel, not only must the power consumption per ton be reduced to a minimum but the consumption of carbon electrodes must be kept as low as possible. The relative economics of graphite, amorphous carbon, and Soderberg paste electrodes may need further study, and some work is being done on coatings for the electrodes which may protect them from oxidation.

### Conclusion

In an article such as this it is only possible to make the briefest mention of the numerous problems where the specialised knowledge of the chemical engineer should help the iron and steel industry in its future development, but it is of interest to observe how the principles of chemical engineering are used under conditions which differ widely from those to which most members of the profession are accustomed.

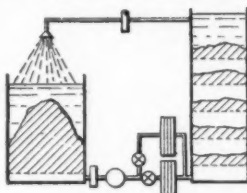
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- <sup>2</sup>*Iron & Coal Tr. Rev.*, 1953, **166**, 179.
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- <sup>14</sup>R. P. Heuer, *Brit. Pat.* 499,302.
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- <sup>18</sup>C. F. Ramseyer, *Iron & Steel Engineer*, 1951, **28**, 57.
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## British Patent Claims

### Precipitating copper

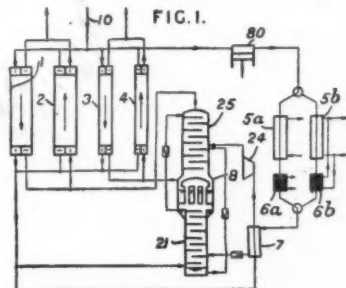
Copper and tin are separated in a process in which tinned copper is treated with an acid solution of copper sulphate, such as spent pickling liquors, under such conditions that all or substantially all of the tin associated with the copper replaces copper in the copper sulphate solution, while the



copper is precipitated as metal. The tin may be recovered from the solution as hydroxide by oxidation with atmospheric oxygen, preferably by spraying through air, and the process may be a cyclic one in which acid copper sulphate is circulated through a batch of tinned copper, a source of copper such as copper scale (to replenish the copper sulphate), atmospheric oxygen (as a spray) and a filtering arrangement for removing tin hydroxide.—698,285, *British Insulated Cables Ltd.*

### Separation of air

In this invention, provision is made for the cooling of a gas mixture, *e.g.* air, and removal of readily condensable impurities therefrom, *e.g.* carbon dioxide and ethylene, prior to separation in a double column 8. This is effected by dividing the air into main currents which are cooled and purified by passage throughout the whole length of alternating heat exchangers or cold accumulators 1..4. These are traversed also throughout their lengths



by reversely flowing cold separated nitrogen and oxygen and into a subsidiary current or currents from which impurities are absorbed, *e.g.* by passage through alternating silica gel containers 6a, 6b.

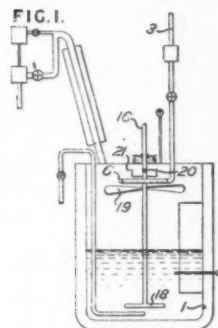
The subsidiary current comprises a small percentage of the mixture entering an inlet 10 and, after compression to 15 atmospheres at a compressor 80, is cooled in alternating driers 5a, 5b. These, together with the adsorbers 6a, 6b are cooled by an ammonia circuit; the current traverses a liquefier 7 before entering a high-pressure column 21.

A major portion of the cold main current enters the column 21 and the remainder traverses the liquefier 7 and an expansion turbine 24 before entering the low-pressure column 25.

In a modification the subsidiary current branches from part way down the cold accumulators before traversing the adsorbers, and the latter are regenerated by warmed returning nitrogen.—697,327, *Ges. fur Linde's Eismaschinen Akt.-Ges.*

### Mixing appliances

A device for intimately mixing air and steam for subsequent chemical reaction with a liquid comprises an



inlet pipe 3, Fig. 1, for air under pressure, terminating in a hollow perforated ring 6. The rotating propeller 19 ensures that the air issuing from the holes in the ring is well mixed with the steam arising from the heated liquid in the autoclave 1. It is sucked into the hollow shaft 16 through the hole 20, the cup 21 preventing un-mixed gases from entering 20.

The mixture passes down the hollow shaft and issues through the cross-piece 18 whence it rises through the liquid in the form of fine bubbles.

In an alternative form (not shown), the air and steam are mixed by a propeller in a chamber external to the autoclave and pass in through a pipe separate from the rotating shaft which is solid and carries a further propeller instead of the tube 18.—695,301, *Monsanto Chemical Co.*

# Electricity in Chemical Factories

By G. Nonhebel, B.A., B.Sc., F.R.I.C., F.INST.F.\*

*At the British Electrical Power Convention at Brighton on June 30, Mr. Nonhebel presented an excellent review of the use of electricity in the chemical industry and discussed some special problems. He raised a great number of interesting points for chemical plant managers and plant designers, as well as the electrical industries, to consider. Some of these appear in the following extracts from the paper.*

## MOTORS AND STARTERS

A wide variety of electric motors and their control gear is used in the chemical industry. The great majority of motors operate under clean conditions and are of normal pattern. By far the largest number of units used are medium-sized (5 to 25 h.p.) motors driving pumps, fans, compressors, pulverising machines, conveyors and agitators. These are generally continuously loaded and reliability is all-important. Sudden failures, for example, of drives to agitators and cooling-water pumps can be very costly. Squirrel-cage induction motors are now so reliable and easy to control and maintain, and the overall energy efficiency from fuel is now so high, that electric drives have in recent years displaced many steam drives, except where there is an economic case for back-pressure steam drives. The greatest use is for totally enclosed fan-cooled motors.

Although many variable-speed a.c. commutator motors are used, they have often been installed when a more economic method of control of the process variable, for example a gas rate, was available. They have also at times been installed for unsuitable duties—for example, when full power is required at low speeds, or in corrosive atmospheres in which the commutators are attacked. Their maintenance charges can then be slightly higher than those for slip-ring motors with continuously rated control gear. Many design engineers prefer squirrel-cage induction motors with variable-speed gear boxes for powers up to 25 h.p.

Where it is regarded as essential that the speed range should be widely variable, d.c. motors are often used. They have some value in new process plants where it has not been possible to predict from small pilot plants what should be the optimum speed. These motors will probably become more popular for variable-speed work in selected areas of a factory, following on developments of compact rectifying equipment and electronic field control

devices. For both a.c. and d.c. motors, there remains the need for adequate cooling when running for prolonged periods at low speed and for metals for commutators which will not be corroded by chemical factory atmospheres. Impregnated brush 'pig tails' are available which do withstand many chemical atmospheres. If the motors are cooled by forced draught of clean air, this difficulty and overheating are both avoided.

Submersible pumps and motors in wells are well known, but in these the motor is 'canned.' An interesting development is, however, a squirrel-cage motor to run 'naked' in an organic chemical medium. As an example, I quote the case where a condensing material is agitated under high pressure at constant speed (1,000 r.p.m.); the motors are specially designed with long, narrow rotors fitted closely in the field structures of the machines which are 'mush' wound. The whole assembly is mounted within the pressure vessels and is immersed in the medium. This arrangement avoids a troublesome gland on the vessels, although at first trouble was experienced with the motor bearings.

## RECTIFICATION

The principal use of direct current is in electrolytic processes, of which the most important are the production of chlorine and caustic soda by electrolysis of brine, and of chlorine and sodium by electrolysis of molten salt. The demand in a single factory may be over 200,000 amp. at 250 v.

The older plants were supplied by steam-driven d.c. generators and some modern plants have diesel-driven d.c. generators. Rotary and motor converters are also used extensively. Mercury arc rectifiers are not so commonly used in Britain, as they have a low efficiency at voltages much below 600 v. and occupy a large floor area. The hermetically-sealed steel-tank rectifier is to be preferred. Rectifiers of the glass type have the disadvantage that highly toxic mercury vapour is

\*Imperial Chemical Industries Ltd.

discharged when the bulb is cracked, as can happen by maloperation, for example by cross-connection. A development of the last few years is the contact rectifier, which was invented abroad, but is now manufactured in the U.K. As its efficiency is over 97%, compared with 92 to 93% for motor converters and 95 to 96% for mercury arc rectifiers at 600 to 700 v., it is now being used in place of the latter types for heavy loads up to 350 v. The commercial development of germanium and silicon rectifiers is awaited with interest.

Ripple in d.c. supplies is said to have some disadvantages in certain electrolytic processes and yet is believed to be beneficial in others. Since methods of smoothing ripple are well known, the onus is on the chemical manufacturer to choose the type of rectification best suited to his purpose.

## PROCESS MEASUREMENT AND CONTROL

There is a strong tendency now to develop electrically operated controlling units to provide more flexibility than is available from the existing pneumatic designs—which have, it would seem, reached very nearly their ultimate performance. There is no sign, however, of a suitable flameproof electrical mechanism for operating large control valves at a cost comparable with that of the pneumatic equipment. There is no doubt that the development of such equipment would be of very great advantage, provided it were considerably faster in response than the present pneumatic equipment.

While electrical equipment has much in its favour compared with pneumatic equipment, there are circumstances in which it suffers seriously from voltage and frequency fluctuations in mains supply. It is not difficult to stabilise the voltage of a d.c. supply or of an a.c. supply in the absence of frequency variations; it is difficult, however, and consequently expensive, to do so when frequency variations occur simultaneously. Therefore, most instrument makers design their equipment to



avoid as far as possible the necessity for a stabilised supply by employing null-balance techniques or, in the case of amplifiers, negative feed-back systems. Few instruments are seriously sensitive to frequency variations alone. Frequency variations are, therefore, only of consequence normally in that they destroy the efficiency of voltage stabilisers.

#### ELECTRICAL COMPUTERS IN PROCESS CONTROL

Perhaps the most important contribution of electrical instruments to process efficiency will be in the application of electrical computers to process control. Much has been said about the 'automatic factory' over the last 20 years, but most of this has not been based on very solid grounds. It is only since the development of the necessary components that have made the electronic computer possible that there has been any real means of designing a completely automatic factory in the process industries. Even now the problems to be overcome before such a factory can be built are many and difficult; it is unlikely that such a factory will be built during the next 10 years. Nevertheless, the electrical engineer can accelerate progress towards this end by providing the process engineer with measuring and control equipment which is more rapid in response, more consistent in use and—above all—absolutely reliable. Already there is one plant with a capital cost of £1 million which is run by only one shift man in the control room and the only other process labour about is employed on cleaning; there is, of course, the usual complement of men employed on maintenance, including the servicing of instruments.

#### SPECIAL ELECTRICAL PROCESSES

The really big unit consumers of power are furnaces for the production of carbide, phosphorus and graphite, all of which use a.c. current and, as already mentioned, electrolytic plants using d.c. for production of chlorine, together with caustic soda or sodium metal. Direct current is also used in the crucible arc furnaces used for refining the commercially novel metal, titanium. All these processes have the problem in common of transmitting extremely heavy currents at low voltage. Thus a typical carbide furnace requires 100,000 amp. at 100 to 150 v., and a row of 50 electrolytic cells in series may require 50,000 amp. at 250 v., though the complete plant may take up to 200,000 amp. The current

densities employed depend on the cost of power and the price of copper, and in recent years lie in the region of 600 to 700 amp./sq.in. of busbar. Argon welding is employed in jointing busbars in order to secure low resistance at the weld and the bars are supported on stout insulators which may be pivoted to allow for expansion. Some bars are hollow and mechanically ventilated to minimise heating.

Heating of chemicals by infra-red emission from appropriately designed electric- (and gas-) heated radiators is making steady progress, but is probably meeting with some resistance from the older branches of the industry. It is an attractive form of heating, since the temperature of the material in process can be raised to the required level in a few minutes, compared with perhaps an hour with hot-air heating; uniform temperatures up to 750°C. can be readily obtained and accurately controlled and, under ideal conditions, the efficiency of energy utilisation can be up to 50%. As might be expected, therefore, infra-red heating is popular in the plastics industry, especially for curing and softening, and in packaging operations in the pharmaceutical industry, for example shrinkage of plastic seals.

Dielectric heating of chemicals by radio-frequency induction has the merit that a bulk of static material can be heated uniformly. As far as I know, there are few plants of this kind in the chemical industry, but further applications are under consideration. Heating of metal vessels containing chemicals by induction at mains frequency is also in satisfactory operation.

On a lower plane, immersion heaters for small still boilers have many applications, and the gases to many catalytic converters are heated by large-bare-wire, electrical heating coils until auto-thermic conditions are reached in the converter-interchanger assembly. Electric trace heating of pipelines conveying hot viscous liquids is well-established practice.

#### LIGHTING

When the atmosphere is heavily charged with corrosive vapours, the problem of maintaining good lighting is serious and special fittings have to be designed; an example is a fluorescent lighting fitting in which all the metal parts are shrouded in PVC.

In explosives factories, and in those where there is a dust-laden atmosphere, pressurised fittings are often used. Pressurised flameproof fluorescent lighting fittings have given satisfactory service in explosive-filling

buildings, but special precautions have to be taken with pressurised incandescent lamp fittings to obviate danger from heat evolved by the lamp. These lighting systems have been developed to meet situations in which electric lighting fittings have been prohibited in the past and by using them it is not only possible to dispense with flame-proof fittings but even to extend the lighting installation to areas with Group IV hazards. Air, nitrogen or carbon dioxide at a pressure of a few inches water gauge is pumped through the lighting installation; pressure relay protection is essential.

On the other hand, some plant managers do not look kindly on pressurised systems, since the arrangement involves more maintenance problems. In one factory, 'authorised' incandescent lamp fittings are installed in the roof of the building so that the fittings are external to it, their cover glasses forming part of the ceiling. The fittings are in a light frame which would blow out in the event of an explosion and thus serve as a relief vent. Resort is sometimes made to the use of fluorescent U tubes passing through glands in the outer wall of the building to terminals outside.

#### FACTORY GENERATION OF ELECTRICITY

The chemical industry is a large user of low-pressure steam for process heating purposes, usually at pressures between 5 and 20 p.s.i. but occasionally up to 150 p.s.i., and consequently there is great scope for back-pressure generation in both large and medium-sized factories. The largest factories generate steam at 800 or 900 p.s.i. and some have on order boilers for generation at 1,700 p.s.i. and 1,050°F. The steam from these high-pressure boilers is used to generate electricity in back-pressure, sometimes mixed back-pressure, and pass-out turbines with relatively small condensers.

In the older factories, large quantities of back-pressure steam at up to 250 p.s.i. are used for directly driving large compressors and pumps, but, with the increased efficiency of electrical generation and reliability of electrical distribution systems and variable-speed motors, it has become the practice to install electric drives, except in the few places where utmost reliability is required or where steam drives are to be preferred to electric drives on account of fire risks. In these latter cases, the steam drives are pass-out turbines for powers above 500 h.p. and pass-out steam engines for smaller powers. It may be taken,

in fact, that introduction of modern electrical drives with the ease of maintenance by motor substitution and the ease of remote control, especially if desired by instrumentation, coupled with the necessity for reducing the cost of manning plant, will lead to a continuing increase in the ratio of electricity to process steam consumption.

A recent survey of industry showed that over half of the industrial boilers in service were over 30 years old, a quarter being over 40 years old. Replacements are therefore inevitable at an increasing rate and will perhaps be assisted by the Government loan scheme for fuel-saving projects. Many of the small factories generate and distribute steam at 100 to 180 p.s.i., though the majority of the steam is used at pressures of only 10 to 20 p.s.i. Hence, when the boilers are replaced, there ought to be an examination of the economics of back-pressure generation. For works with process steam demand of 20,000 to 50,000 lb./hr., the right replacement in my opinion is usually the *Economic* boiler, because of its low cost and small ground space and height requirements. Although the highest pressure available with these boilers is 250 p.s.i., which means 240 p.s.i. at turbine stop valves, sufficient back-pressure electricity can be often generated, if the process steam pressure is kept low, to justify economically the installation of new boiler plant, coupled with back-pressure plant. For larger steam demands, water-tube boilers become superior to shell boilers, but, in general, the pressure should be raised to over 250 p.s.i., and suitable pressures would be either 400 or 650 p.s.i. At least one boiler manufacturer has now developed a compact small water-tube boiler which is suitable for chain grate or oil firing and is competitive with the *Economic* boiler.

It is not necessary for the back-pressure generator always to be at the boiler house. In one case recently a new back-pressure set has been installed in an old factory several hundred yards away from the boiler house but adjacent to the main sink for the process steam. In another case, there has been installed in parallel across a pressure-controlled reducing valve to a process heating vessel a simple turbine driving a low-voltage induction generator. No special protective gear is provided. This system may horrify the orthodox, but appears to be economic. It is doubtful if either of these cases would be economic in a new factory with H.P. boilers, since L.P. steam mains, despite their bigger diameter, are no more expensive

than H.P. mains and a great advantage is to be gained by keeping steam and power generation together with distribution control under one roof.

#### SAFETY

There has always been some controversy throughout the chemical and oil industries on the most suitable electrical drives in dangerous areas. In Britain, the oil industry regard flameproof motors as satisfactory provided they are well maintained, but in some oil refineries abroad flameproof motors are difficult to service with the semi-skilled labour available, and totally enclosed but forced-ventilated motors, specially designed to avoid sparking, are installed for most duties; this arrangement is being considered for parts of many refineries in this country. No electric motor has so far been certified for Group IV conditions and consequently steam drives for pumps and fans become a necessity.\* In many situations safety is achieved by pressure ventilation of totally enclosed motors with air drawn from a safe situation. Similarly, control rooms in danger areas are now often 'pressurised' because of the potential sources of sparking in the instruments. In other situations safety is achieved by placing motors outside the walls of the building with shaft drives through stuffing boxes to equipment inside. Similarly, motors and their push-button controls may be flameproof, but their control switchgear may be placed in a 'safe' area and therefore be of normal industrial type.

Another point that requires emphasis is the need for safe dustproof motors for use in areas where dust explosions could arise. The flameproof motor is not dustproof. In the absence of a B.S. specification, H.M. Chief Inspector of Explosives has issued a memorandum setting out the conditions which he regards as meeting the situation, but it follows that further research and development by the industry is required on this point. In the meantime, I note that the Electrical Research Association has started a study of the criticisms received from industry on flameproof equipment and will be starting a study of design requirements for dustproof equipment.

It will be seen from the above that, especially from the safety point of view, the greatest care and collaboration is required between the electrical engineer and chemical plant designer.

\*The opinion is, however, held that motors for Group III could be used where the only danger is of escapes of hydrogen, provided the building is well ventilated.

### The Leonard Hill Technical Group—August

Articles appearing in some of our associate journals this month include:

**Manufacturing Chemist**—Adrenergic Inhibitors, 1; Classification and Structure; Tablet Making; Hydroponics; Perfumery Chemicals; Progress Reports; Pest Control Chemicals, Perfumery and Essential Oils.

**Food Manufacture**—The Dehydration of Cabbage; The Chemistry of Peanut Butter; Old-Time Potted Meats; Food Investigation Organisation Report; Lecithin in Baking; Processed Foods from Jamaica—New Standards Devised; Food Research at Leatherhead; New Stork Margarine Laboratory; Institute of Food Technology Abstracts.

**Paint Manufacture**—Thixotropy and Paints, 4; Chromatography of Natural Resins; New Analytical Technique; Paint Manufacture in Belgium; Progress in Printing Inks; Report of the PATRA Exhibition; Reports on the O.C.C.A. Conference, British Instruments Industries Exhibition, the Institute of Metal Finishing conference, the Colour Congress at Heidelberg and the opening of new I.C.I. paint development laboratories.

**Fibres**—Industrial Uses of Artificial Fibres, 2; Second International Textile Exhibition: Report of Brussels\* Exhibition; 40th Annual Conference of the Textile Institute: Practice and Progress; The Drying and Heat Treating of Textiles Containing Cellulose Fibres; Arnel Triacetate Fibre.

**Atomics**—The Angular Correlation of Prompt Fission Neutrons; The Derivation of Maximum Permissible Levels of Contamination of Surfaces by Radioactive Materials; The Chemical Effects of Ionising Radiation in Solids, 2; British Equipment at Geneva, 1.

### Petro-Chemicals Expansion

(Concluded from page 290)

being designed and built by Stone & Webster and is scheduled to begin operation in the last quarter of 1955.

With a few specialised exceptions, all the equipment for the new plant will be purchased and fabricated in the U.K.

All the off-site facilities are being designed and constructed by the British Petroleum Co. Ltd., who are acting as managers of the whole project.

As with the original plants, steam and electricity will be provided for the extensions by Grangemouth Petroleum Refinery Ltd. The local authority is making arrangements to meet the increased demands for water.

An increase of approximately 250 in the labour force will be required.

# Plant and Equipment

## Pumping powders

The 'DM'-type pump for handling dry materials, Mono Pumps Ltd. inform us, has completed its stage of development which entailed extensive field research. In consequence two sizes of the 'DM' type are now available for handling loose dry powders devoid of clogging, compacting or fusing tendencies. The DM12 unit is capable of outputs up to 250 cu.ft./hr. when running at a speed up to 300 r.p.m. Horse powers of only 2 to 5 are required, depending on the physical characteristics of the powder, the degree of fluidisation and on the nature of the piping system.

The DM10 size is available for outputs up to 100 cu.ft./hr., using running speeds up to 250 r.p.m. Fluidisation of the powder is necessary to reduce friction in the pipeline. For this purpose the use of air is kept within very low limits.

## Steam traps

Prototypes of a complete new range of ball-float steam traps in three series were shown at the annual engineers' conference of Spirax-Sarco Ltd. which was held in Torquay recently and attended by associates, representatives and technical personnel from home and abroad. The three series will gradually replace the corresponding sizes in the

present series of Spirax ball-float traps, covering the existing full range of steam-pressure requirements and, in certain of the sizes, giving greater condensate discharge capacities. For example, the FT 700 series will give discharge capacities up to 10,000 lb./hr. and the FT 800 series as great as 40,000 lb./hr.

Design features of the new steam traps include easily interchangeable units for thermostatic air release and steam-lock release according to the conditions and requirements.

The Spirax IB series of inverted-bucket steam traps, of new and unorthodox design, and the redesigned Spirax Ogdan open-top bucket steam traps are complete and the full ranges of both types are now being marketed.

Among the developments in thermostatic control equipment announced, an item of special interest is a new three-way valve for the automatic control of diesel engine and compressor cooling water temperature.

A predominant feature of the technical application sessions of the conference was the growing contribution of heat control and heat-transfer methods towards increased output efficiency of process steam plant. The discussions ranged over a wide field of heating and process steam usage and particular attention was directed to improved methods of venting air from steam-heated units.

## Filtering slurries

A recent development in filtration equipment is the Eimco Burwell filter, which is claimed to have a number of advantages over the conventional plate and frame filter press. Characteristics of the Burwell are:

(1) Mechanical operation for opening and closing the unit and for removing and replacing the frames.

(2) Two sets of frames so that down time for cleaning is greatly reduced.

(3) Feed entry at the bottom centre of each frame to prevent segregation of the solids.

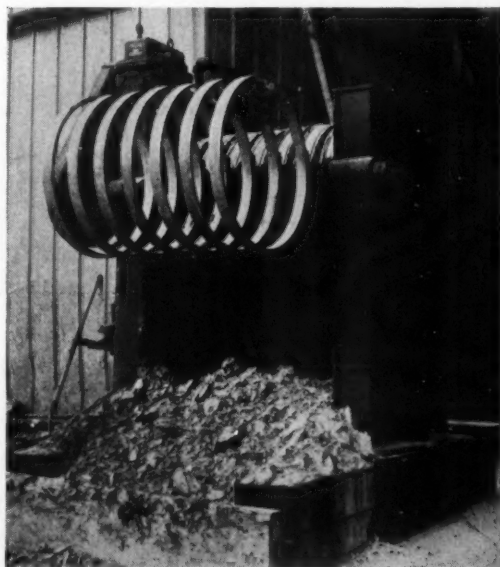
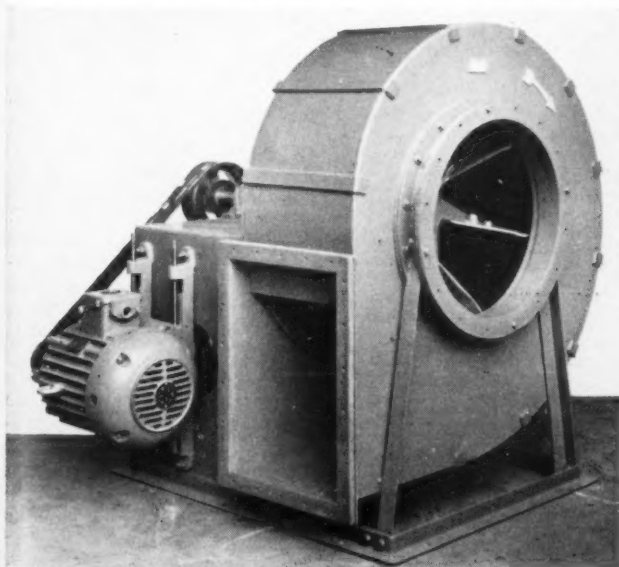
(4) Even cake formation due to the feed inlet position. Two filter cakes, separated by a small gap, are formed within each frame.

(5) Provision for blow-back with either air or inert gas through each filter cloth in order to free the filter cake and prevent the cloth from blinding.

(6) Provision of synthetic gaskets between frames and filter media to minimise cloth wear.

Two sets of frames are mounted at 180° to each other on a shaft which can be rotated mechanically. The plates are mounted on a standard shaft on the opposite side of the filter; they are separated on the rotating shaft by cast lugs.

Sealing of the plates and frames is effected by means of a piston-diaphragm arrangement of the plates, which are male and female, and connected to a compressed-air header. The slurry to be filtered enters from



Left: A large paddle-blade centrifugal fan in rigid PVC, one of a range supplied by Turner & Brown Ltd. Right: The filter described on this page is illustrated by this pilot-scale unit which has been used for filtering tungsten leach residue in the works of the Salt Lake Tungsten Co., U.S.A.



an inlet header through hose connections into the feed channel; the feed channel is made up of a series of mating ports in the plates and frames. Wash water and air-blow are introduced into the same feed channel, but from separate headers.

While the operating cycle is taking place, one set of frames remains in the 'ready' position. At the end of the cycle, the cake-laden frames are rotated mechanically through 180°. Thus the second set of frames is immediately brought into position ready for the next operating cycle which can commence before the cake is discharged from the first set of frames. Discharge of the cake from the full frames can then be effected when the filtering is again on-stream; in practice, most of the cake falls out of the frames as they are swung into the discharge position. By this means an extremely rapid turn-round, of less than 5 min., can be achieved, it is claimed.

This information about the *Burwell* filter is supplied to us by Eimco (Great Britain) Ltd., a subsidiary of the Eimco Corporation in America. The *Burwell* is a very recent development and, unlike most of the British company's range of equipment, is available only for dollar payment as yet.

### **Glass chemical apparatus**

Quickfit & Quartz Ltd. are introducing a number of additions to their standard range. One of these is a steam-heated circulatory cyclone evaporator, giving rapid evaporation either for concentration or recovery. When steam at approximately 2 to 3 p.s.i.g. is passed through the coil, the apparatus will evaporate aqueous solutions at a rate in excess of 5 l./hr. under the vacuum of a water pump. The coil condenser incorporated in the apparatus is of large surface area to ensure that complete condensation occurs. As the apparatus incorporates a cyclone, it is most effective in stopping entrainment of liquids or solids in the vapour phase. Its main advantage is continuous rapid evaporation with a small working volume (approximately 500 ml.). The minimum residual volume in the heater is 300 ml. The makers state that the relatively low temperature of the heat source and short contact time make it suitable for heat-sensitive materials.

Another addition is an all-glass, climbing-film-type evaporator, capable of evaporating aqueous feed stock at the rate of 3 l./hr. when under vacuum, so that water boils at approximately 40°C. A typical use is concentration

of fruit juice at the laboratory stage—with single-pass working, concentration to 25 to 50% is normal.

A further new Quickfit item is a semi-micro molecular still in which the liquid to be fractionated is allowed to run slowly down a platinum or nickel spiral closely fitting inside the glass tubular body of the still. The outside of this is heated electrically by a jacket, the temperature of which is carefully controlled so that the most volatile compound is separated from the remaining liquid. Condensation takes place on a water-cooled condenser tube, which forms the axis of the spiral. On completion of the run, the still is turned through 180° on the spherical joint fitted to the vacuum lead and the process repeated as many times as is necessary to obtain the required fractions.

### **Metering free-flowing solids**

A pneumatic device known as a *Massometer* is described as presenting a new principle in flow measurement—continuous metering of flowable solids by weight. It is stated that changes in the rate of flow—even momentary fluctuations—are detected instantaneously, since each particle being measured is in the metering impeller for only a fraction of a second.

The stream to be measured falls on a rotating impeller driven by a synchronous-speed electric motor which is supported in bearings and therefore free to move about the axis of the motor shaft. The torque on the motor housing, which is created by accelerating the particles of the stream to the tangential velocity of the impeller, is proportional to the mass rate of flow. This torque is measured by a pneumatic prime relay which operates on the 'flapper and nozzle' principle. The output of the pneumatic relay is proportional to the rate of flow of material and is recorded on a circular chart, pressure recorder which reads in lb./min. of material.

Any stream of free-flowing solid material can be weighed instantaneously and recorded on a continuous basis, the makers state. Besides

its uses in materials accounting and in process control, the instrument may be preset to deliver a given amount where it is necessary to add a definite amount of an ingredient to a process or batch. When the integrator totals this amount, a signal can be used to stop the flow automatically.

In addition, the pneumatic output of the *Massometer* to the recorder can be used to control other feeders or regulators. The feed rate of these feeders or regulators will then be proportional to the flow of the main stream measured by the *Massometer*.

Wallace & Tiernan Ltd. are the suppliers.

### **Level control**

A new Mason-Neilan level controller that is being produced by the Crosby Valve & Engineering Co. Ltd. supersedes the series 12600 level controller which the company have been manufacturing for some years. Whilst the fundamental principles are the same, this instrument incorporates many additions and improvements. The pneumatic control mechanism is mounted direct on a cast-iron frame which itself is bolted to the torque tube housing, thus eliminating the possibility of any control offset due to mishandling of the instrument case.

The specific gravity adjustment scale, which enables the instrument to be set for the density of the liquid being controlled, has been widened to a range of 0.5 to 1.4 sp. gr. The set point scale has been considerably increased in length, the method of adjustment is simplified and it is so arranged that a 3 to 15 p.s.i. bellows for remotely setting the control point can easily be provided.

A new and improved reset or integral action resistance unit can be fitted to a standard proportional controller on site. The size of the torque tube mechanism chamber has been reduced, thus saving weight and space. The dimensions between connection centres now conforms with international standards. The new pilot valve uses slack diaphragms of special materials and incorporates a jewel orifice fitted with a plunger to enable this orifice to be cleaned whilst the instrument is in operation. The method of setting the accurately calibrated proportional band has been improved to facilitate setting and locking.

This controller is available for mounting with the displacer either inside or external to the vessel concerned and is supplied as a proportional controller with or without reset, as a

*For further details of  
the plant and products  
described, please use  
the coupon on page 310*

transmitter, as a specific gravity transmitter or controller or as a combined controller and transmitter.

### Constant-pressure pump

The makers of a range of positive-displacement rotary pumps announce a new design which, they state, will enable a constant pressure to be obtained, irrespective of the flow conditions. Although originally developed for gas turbine testing, it is now being used for oil fuel firing, and the makers think it will have extensive uses in the chemical and process industries.

The control is simple and comprises only a small adjustable dead-weight accumulator and an actuating cylinder mounted on the pump body, the latter having a connection to the delivery line and varying the pump output by the differential between the accumulator and the line pressure.

Among the advantages claimed for the new (C.P.C.) system are:

- (1) The burners can be operated at all times at their optimum pressure.
- (2) Burners can be shut down, opened up, or nozzles changed without any readjustment of the pumping system.
- (3) Pressure is stabilised regardless of any changes in fuel viscosity resulting from changes of temperature or fuel characteristic.
- (4) The fuel oil system can be kept at optimum firing temperature and pressure, even when all burners are shut down—giving immediate availability in emergency or for sudden peaks.

(5) The pumps only absorb the power required to deliver the amount of oil being fired, i.e. there is no wasteful by-pass system required.

(6) The C.P.C. system can be instantaneously adjusted to any desired delivery pressure from 10 to 500 p.s.i.

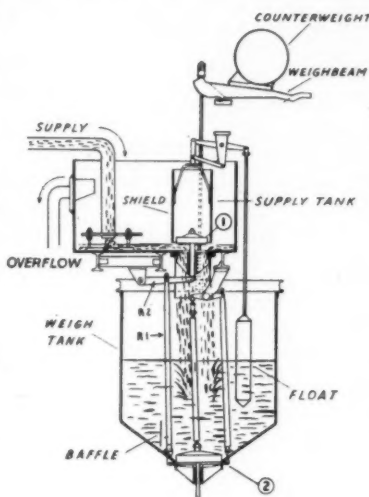
(7) Pumps with the C.P.C. system are available for all fuels from petroleum spirit to bitumen, including C.T.F. (creosote pitch); and in capacities ranging from 100 to 80,000 gal./hr.

The suppliers of the pump are Plenty & Son Ltd. For their general range of pumps, this company have been experimenting with certain plastics, and for some duties they are offering pumps with internal components made of *Railko* or *Fluon*. Another recent development is a pump fitted with electric solenoids instead of the standard hand control which is inter-connected with a weighing machine. This is stated to enable the accurate measurement of weighed quantities of liquids into drums.

### Weighing liquids

Calculation of the weight of liquids from the cubic measurement is often unsatisfactory; a device known as the Fletcher automatic liquid scale is claimed to be an advance on this method. It is stated that it continuously and automatically weighs liquids of all viscosities up to 150,000 ssu., and is suitable for weighing edible liquids, chemicals, spirits, oils, hot or cold water, etc.

It is based on the principle of tare weighing, the operating power being provided by the liquid being weighed. The scale will continue to discharge a given weight of liquid while the supply is maintained. The rate of weighing is controlled by an oil buffer



which works smoothly and without shock. None of the balance parts are in contact with the liquid and thus, the makers state, corrosion and wear are negligible. Exactly the same weight of liquid is discharged at every weighing and an automatic counter registers either the number of weighings or the total tonnage. The settings can be locked and any Government excise seals affixed as may be required.

The Fletcher scale can be fitted with an automatic sampling device and also with a small tank which provides for the automatic addition of a predetermined but variable volume of secondary liquid with each discharge of the weigh tank. The scales are built in four sizes to deal with  $\frac{1}{2}$ , 1, 2 and  $3\frac{1}{2}$  tons of liquid per tip respectively, giving a maximum capacity of 150 tons/hr. of a liquid having a viscosity equal to that of water.

George Fletcher & Co. Ltd. are the suppliers.

### NEW PRODUCTS IN BRIEF

New form of straight, unplasticised PVC, available as rigid pipes, rods and welding wire. Expected to find use in industries whose problems are either the conveying of corrosive liquids or gases, or the operation of pipelines and similar plant in corrosive atmospheres or aggressive soils. Makers claim material is tough; light in weight, hygienic, corrosion resistant, easily worked and formed. (Chemidur Plastics Ltd.)

Stainless-steel bubble-cap trays, 5 ft. 3 in. diameter, have patented wedge-type clamping devices for holding down the bubble caps and tray floor sections instead of the nuts, bolts and washers normally used. (Metal Propellers Ltd.)

Collapsible containers of extra-strong laminated PVC, double-welded by a special process, made in capacities up to 5 gal. Claimed to be corrosion proof and acid resistant, suitable for transport and export of oils, fruit juices, etc. (B.X. Plastics Ltd.)

Moisture meter, claimed to give high accuracy and to be capable of testing from completely dry to 100% water or any moisture content in between these limits. Weighs 10½ lb., measures 10 in. × 10 in. × 10 in., and costs £98 in Great Britain. (Shaw Electronics.)

Special feature of a new steel packaging drum is a valve and screw-bung fitting which forms an integral part of the drum. This is so designed as to provide for the vacuumising of the container after filling, for the subsequent insertion of an inert gas if so desired, and for the maintenance of a 25-in. volume of vacuum. An inexpensive check valve and gauge may be used for periodical check readings without impairing the vacuum, makers state. (Reads of Liverpool.)

Flameproof process control alarm, providing both visual and audible warning of an abnormal process condition. Made as a two- or three-light unit and can be adapted either as a straightforward alarm or for combined alarm, indication and control. Also available—a flameproof single-light unit which can be arranged as a combined push and indicating lamp or alternatively as a combined indicating lamp with stop-start and other switching features. (General Electric Co. Ltd.)

# Coal and Chemicals

## British Fuel Chemists' Latest Discoveries

### Germanium

THE increasing importance of germanium to the electronics industry has led to a study being made of the way in which germanium present in coal becomes concentrated in certain of the by-products during processing or combustion of coal. It is hoped to discover all possible sources of material rich in germanium and to discover ways of modifying processes and plants to increase the amount of germanium which can be recovered.

The distribution of germanium in the coke, tar and liquor produced by the carbonisation of coal is being investigated and the dusts and deposits collected from various points in the flues of a coke-burning producer have been examined for their germanium contents. It was found that about 90% of the germanium present in the coke when it was fed to the producer was volatilised but only a proportion of this volatilised germanium was retained in flue deposits. It is likely that considerable quantities of germanium pass out of the chimney stack in the form of a very fine dust.\*

This is one of the interesting advances that are recorded in the annual report† on the work carried out under the Fuel Research Board (D.S.I.R.). The report, as in previous years, mentions a number of projects that are of interest to chemical engineers and to industrial chemists in various fields and some of these are referred to in the following article. During the year under review additional laboratory accommodation, largely of a temporary nature in view of the projected move to a larger and more suitable site at Stevenage, has been built at the Fuel Research Station at Greenwich to relieve overcrowding and provide more satisfactory facilities for a number of the research programmes. The new buildings include a laboratory for chemical engineering research. A room has also been remodelled with thick concrete shields to allow a 100-curie cobalt-60 source to be used in studies of the effects of gamma radiation on coal and coal products and on the rate and course of some of the chemical reactions of interest in fuel science and technology.

\*See also the item on 'Germanium from boilers' on page 282.

†Fuel Research, 1954, H.M.S.O., 3s.

### Pyrites residues in coking process

As mentioned in the previous report, work has been undertaken at the suggestion of the National Sulphuric Acid Association Ltd. to see whether finely divided iron oxide residues from the flash roasting of pyrites can be made use of by mixing with coking coal before carbonisation for the production of metallurgical coke. The residues are too finely divided for direct use in blast furnaces or for sinter plant. This work has been extended by tests with a coking slack as used in a Lincolnshire ironworks. The results confirmed those obtained in previous tests using Durham and Scottish coals, and again showed that the addition of up to 7% of the iron oxide improves the shatter strength of the cokes produced but increases the abrasability.

### Oils and chemicals from coal

Work has continued on the Fischer-Tropsch synthesis, being primarily concerned with a study of the basic reactions and the development of a more economic form of the process. An important part of the investigations in progress consists in the development of catalysts which combine satisfactory activity and selectivity (ability to direct the reaction towards the desired end products) with low cost, and in establishing the effect of temperature, pressure and gas composition on the yield and composition of the products and on the life of the catalyst.

During the past two years experimental runs in a pilot plant treating 2,000 cu. ft./hr. of gas have been carried out using the fluidised-catalyst system. The results obtained in two runs were summarised in *CHEMICAL & PROCESS ENGINEERING* in March.

Tests in a small, 1-in.-diameter, fluidised-catalyst reactor with samples of catalyst withdrawn from the pilot plant confirmed the observations, previously reported, that the performance in the small unit is significantly better than in the large reactor. This is in agreement with the observation of other workers that reaction rates in fluidised beds tend to decrease with increase in the diameter of the bed. It would appear, therefore, that if a full-scale Fischer-Tropsch unit is to reproduce the performance obtained in the pilot plant it is essential to avoid any marked change in the geometry of the bed.

### Liquid-phase ('slurry') process

Investigations of the liquid-phase process are concerned not only with the development of suitable catalysts and the determination of the effect of the normal reaction variables such as temperature and pressure, but also with studies of the mechanical aspects of the system to assess the significance of such factors as the geometry of the reactor, gas distribution, bubble size, and stability of the catalyst suspension. These aspects are of particular importance in translation of the process from the experimental to the full scale.

It has been found, following American experience, that the stability of the present catalyst-in-oil suspension is improved by adding 1 to 2% of an organophyllic clay, 'Bentone 34,' as an anti-sedimentation agent. There is some evidence from experiments in which the ratio of recycle gas was varied, that performance may depend on attaining an optimum degree of turbulence with a suitable linear gas velocity and expansion of the gas-slurry mixture. The physical nature of this mixture is determined to a great extent by the gas distribution system at the inlet to the reactor. A series of experiments, using glass tubes of diameter 1 to 8 in., has been carried out to find the best distributor for use in the slurry pilot plant now being erected. The most efficient distributor so far devised consists of a vertical sintered metal 'candle' (or multiplicity of candles) which gives a uniform mixture of gas bubbles and liquid occupying a volume twice that of the settled liquid at low linear gas velocities. Such expanded mixtures have not so far been obtained when the gas is introduced, as in the present laboratory-scale plant, through a relatively coarse porous plate or through a jet. Investigations are being made of the ability of expanded systems to maintain catalyst particles in stable suspension, and the effectiveness of the suspensions for synthesis.

As the synthesis reaction proceeds, the volume of slurry gradually increases by accumulation of high-boiling product. It is necessary, therefore, to remove part of the liquid of the slurry at intervals without removing the catalyst in suspension. After some initial difficulty, a satisfactory method of controlling the volume of the slurry has been achieved by withdrawing part



of the liquid through a filter system operated under a controlled and small difference of pressure over the filter.

Preliminary results from the operation in the liquid-phase of catalysts prepared by the decomposition of alkali-metal ferrites and reduced in the slurry with hydrogen at 20 atm. have been disappointing. The performance obtained has been no better than that of mill-scale catalyst when the reaction rate is calculated on the volume of slurry although it is much superior when related to the weight of iron present. Research on ferrite catalysts, however, has been complicated by an unexpected phenomenon, the conversion of the mobile slurry to a gel which does not flow freely at reaction temperatures and which has made operation impossible unless very dilute slurries are used. This effect, which occurs soon after synthesis is started, appears to be associated with organic material, possibly a complex containing iron, strongly adsorbed on the catalyst.

It has been established for sintered and fused iron catalysts that, in general, granules of size about 10-mesh B.S. test sieve, require reduction to free metal before exhibiting activity for the Fischer-Tropsch synthesis. It has now been found, however, that an unreduced mill-scale catalyst, ground to a mean particle size of less than 10 microns, can be used without pretreatment for synthesis in the liquid phase at 265°C. and 20 atm. During the first few hours, the activity increases to a constant level which is about 70% of that attained by the same catalyst when reduced in hydrogen. The composition of the products was markedly different from that obtained with reduced catalyst, formation of methane being only one-third as high and the proportion of wax (b.p. > 300°C.).

Investigations of the reaction mechanism has included studies based on the composition of the products and studies based on radio-active tracers, while other work has been concerned with the pore structure of catalysts.

### To Authors

*The Editor welcomes practical articles and notes on chemical engineering and related subjects with a view to publication. A preliminary letter or synopsis outlining the subject should be sent to The Editor, CHEMICAL & PROCESS ENGINEERING, Stratford House, 9 Eden Street, London, N.W.1.*

## Personal Paragraphs

★ **Dr. J. W. Barrett** has been appointed a director of Monsanto Chemicals Ltd. He first joined Monsanto in 1941 as a group leader in the then research department. He was



appointed assistant director of research and development in 1950 and later in the same year became general manager of the development division. In 1954 he was appointed a member of the company's executive committee. Dr. Barrett, who is also a director of Monsanto (Soil Conditioners) Ltd., Monsanto Plastics Ltd. and Monsanto Phosphates Ltd., will continue his present responsibility for the company's research and development activities.

★ **Lord Clitheroe** has been appointed deputy chairman of Borax Consolidated in place of the late **Mr. James Gerstley**.

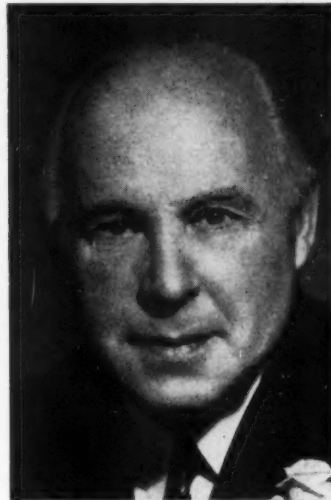
★ **Mr. W. E. V. Abraham** has resigned from the board of British Petroleum Co. and **Mr. W. E. Eadie** has been appointed to fill the resulting vacancy.

★ Executive vice-president **Mr. William P. Drake** has succeeded **Mr. George B. Beitzel** as president of the Pennsylvania Salt Manufacturing Co. (U.S.A.).

★ **Mr. P. J. A. Turner**, who is chief engineer at the company's Glasgow factory, has been appointed a director of Kelvin & Hughes (Industrial) Ltd.

★ **Mr. C. G. F. Pritchett** has taken over the position of chief engineer to Chloride Batteries Ltd. in succession to the late **Mr. C. P. Lockton**.

★ **Sir Roger Duncalfe**, chairman of British Glues & Chemicals Ltd. and president of the British Standards Institution, was elected president of the International Organisation for Stan-



dardisation at its triennial general assembly in Stockholm. He is well known for his services to industrial standardisation, for which he was knighted in 1951. In addition to his chairmanship of technical committees on standards projects, and of the Chemical Divisional Council, he is a past-chairman, past-president, and vice-president of the Association of British Chemical Manufacturers; a vice-president of the Federation of Gelatine and Glue Manufacturers; a vice-president of the Federation of British Industries, and chairman of the F.B.I.'s technical legislation committee. He was also deputy-chairman of the Beaver Committee on air pollution.

★ **Mr. R. J. Glinn** has retired after 44 years with Babcock & Wilcox Ltd. He has also retired from the board of the Calorizing Corporation, of which he has been a director since 1928. He has been responsible for much pioneering work on oil firing, on pulverised fuel, and on the development of automatic soot blowers.

★ **Edwin Danks & Co. (Oldbury) Ltd.** announce the retirement of **Mr. R. C. Nash**. He joined Babcock & Wilcox in 1910 and transferred to Edwin Danks in 1924 to take charge of the estimating department. He was appointed chief assistant to Mr. J. Smith, the managing director, in 1943.

# World News

## GREAT BRITAIN

### Vinyl acetate project

British Celanese Ltd. announce that a plant for a substantial production of vinyl acetate monomer is now under construction at their Spondon works. The plant is designed to operate a process, developed by the Celanese group of companies, starting from acetaldehyde and acetic anhydride. These materials, which are already produced in adjacent plants in very large quantities from products of the company's oil cracking installation, are also used by the company as raw materials for their cellulose acetate production and other manufactures.

The British Celanese Ltd. plant will be the only one in Great Britain producing vinyl acetate by this route, although the process is being successfully operated in America by the Celanese Corporation of America. It is expected that 'Celanese' vinyl acetate monomer will be available in bulk quantities in the early months of 1956.

In his annual review, the company's chairman stated that initially the plant would be capable of producing the equivalent of one-third of present domestic consumption (see page 280).

### PTFE plant's progress

Coinciding with our article on PTFE comes the news that Imperial Chemical Industries Ltd. are making good progress with a new plant at Hillhouse, Lancashire, for the production of *Fluon* polytetrafluoroethylene. When completed, this will have a capacity of about 200 tons, which will be achieved in two stages. The first stage, due in January, 1956, will mean that annual production of polymer is stepped up from 25 tons to 100 tons. The final 200-ton stage should be reached later in 1956. It is also planned to extend the present range to include an extrusion grade and special dispersions.

I.C.I. hopes to reduce the price of *Fluon* later this year or early next year.

### Dunford & Elliott subsidiary

Dunford & Elliott (Sheffield) Ltd. announce that the department of their company now operating from Linford Street, London, S.W.8 is being formed into a separate subsidiary company. The new company is to be styled Dunford & Elliott Process Engineering Ltd. This company will handle the rotary louver dryers, coolers and

roasters and other steadily expanding activities which are indicated by the new designation.

### Steam plant for atomic power projects

Contracts for extension to Britain's atomic power supply system amounting to several million pounds, have been placed with Babcock & Wilcox Ltd. who supplied the steam plant for the original station at Calder Hall. The new contracts cover what is in effect three new Calder Halls. Eight steam towers each 80 ft. in height, will be supplied to a new station to be known as Calder Hall 'B.' Two further installations totalling 16 towers, will be supplied to a power station at Chapelcross near Annan in Dumfriesshire.

### Crushing plant contract

The G. & T. Earle Group of the Associated Portland Cement Manufacturers Ltd. have placed with Fraser & Chalmers Engineering Works contracts for limestone crushing plant and a shale crushing plant, for their Cauldon project in Staffordshire.

A Dixie moving breaker and back plate hammer mill is being supplied for the shale crushing plant. The mill is direct coupled to a 300-h.p. motor and weighs some 34 tons. The machine is of a type whose general application is for the secondary crushing of sticky rocks and ores. Also included in the contract are a hopper, apron feeder, supporting steelwork and chutes.

For the limestone crushing plant a 72 in.  $\times$  19 ft. heavy-duty rubber-mounted pan feeder is on order. This is stated to be of such design that it will withstand the shock of stone falling direct from 15-ton dumper trucks. A hopper, structural steel, chutes and access platforms are also included in this contract.

### Fertiliser Society's growth

At the eighth annual general meeting of the Fertiliser Society, held in London recently, Dr. R. Stewart was elected president and Dr. J. Manning vice-president.

Dr. B. Raistrick, Dr. E. M. Reid and Dr. D. Williams were elected to fill the three vacancies on the council.

The retiring president, Mr. J. T. Procter, referred to the continued growth of the Society and pointed out that its extending overseas membership now accounts for 18% of the total and is drawn from 22 different countries. He stressed, in particular, the inter-

national character of the general meeting to be held that day.

### New pipeline contract

Having just completed the 348-mile, 16-in. o.d. natural gas line from Sui to Karachi in 5½ months, Pakistan Constructors, the joint venture of D. & C. & Wm. Press Ltd. and Morrison Knudsen Co. of Pakistan have been awarded the contract for an additional 188 miles of the same size pipeline running from the Sui gas fields to Multan, later to be extended to Lahore with branch lines to Kot Addu, Montgomery, Jhang and Maghiana.

Work on this line, which is identical with the Sui-Karachi line, will start in mid-1956, completion being expected within six or seven months and estimated to cost \$13,791,540.

The country in which the contractors will work is arid and particularly difficult, there being no rail or road communications. One major crossing of the Indus that will be involved is estimated at 5 miles.

### Engineering work for nuclear power station

John Thompson Ltd., Wolverhampton, has been entrusted by the United Kingdom Atomic Energy Authority (Industrial Group) with a major engineering contract in connection with the Dounreay nuclear power (fast breeder reactor) station.

The work involves the development, in collaboration with the Atomic Energy Authority, of methods of fabrication for the liquid metal circuits, the design of complete steam and water equipment, the manufacture of the whole of the fusion-welded stainless steel vessels, liquid metal pipelines, primary and secondary heat exchangers, boiler drums and of the associated plant, valves and instrumentation, together with the feed-water treatment plant. The company has developed a separate department to deal with this contract, and manufacture has commenced.

### Vacuum Oil in South Wales

A new South Wales Industrial Division of the Vacuum Oil Co. has been formed, with headquarters at Cardiff.

### New company

The Coniston Engineering Co. Ltd., has been formed to carry on business as designers of heavy projects and process plants. The new company will take over the business of the Coniston Co. at Wimbledon which will continue under its present name.

## GERMANY

### Potash expansion

The West German potash industry plans to continue expansion of production installations this year in view of good prospects for domestic and foreign sales, a spokesman has stated recently. An increase in the world demand for potash was expected this year and would help the industry.

German production of potash rose last year to 1,617,870 tons in terms of  $K_2O$ , accounting for about a quarter of the total world production against 1,323,864 tons in 1953.

Production in the first quarter of the year rose steadily. In January it was 146,710 tons, February 147,441 and in March 159,377 tons. Exports accounted for 40% of output in 1954. Main foreign buyers were Britain, Denmark, the Netherlands, Belgium and Japan. Due to an increase in domestic consumption to 830,000 tons in 1953-54, stocks at the end of 1954 were relatively small at 130,000 tons.

The five companies engaged in potash production in West Germany are the Vereinigte Kaliwerke Salzdetfurth A.G. at Hanover, the Wintershall A.G. at Kassell and Celle, the Burbach Kaliwerke A.G. at Wolfenbuettel, the Kali Chemie at Hanover and the Kaliwerke Buggingen at Baden. These companies have invested a total of 550 million marks in the last seven years for rebuilding and expansion.

### Aluminium expansion

The output of primary aluminium from West German plants rose to nearly 130,000 tons in 1954, which establishes Germany as the most important producer of this metal in Europe and third only to the U.S.A. and Canada as a world producer. Production in 1955 is expected to

reach between 135,000 tons and 140,000 tons although it is expected that the actual consumption of aluminium will rise to some 165,000 tons.

## CANADA

### A.P.V. in Canada

The A.P.V. Co. Ltd. have formed a new Canadian company, A.P.V. (Canada) Equipment Ltd., to take over Walker-Wallace Ltd., Toronto, the dairy engineering company who have been their Canadian agents since 1929.

It is intended to extend the scope of the new company to cover the sale of equipment to the brewery, food, chemical and related industries in addition to maintaining the existing manufacturing facilities in Toronto.

### Canadian potash plans

Western Potash Corporation Ltd. has arranged \$17½ million of financing for development of its Saskatchewan potash reserves. The financing group will provide all means for placing the property in production, with initial output expected to be 1,000 tons/day of finished products.

## SPAIN

### Chemical firm's expansion plans

The Union Espanola de Explosivos is to expand its output appreciably by new installations at its works at Seville. It is planned to raise superphosphate production by 120,000 tons p.a. and also to produce 31,500 tons of sulphuric acid. Total value of machinery for the new installations is placed at \$85½ million, of which part will be manufactured in Spain and part imported.

## INDIA

### Conserving coking coals

The Council of Scientific and Industrial Research of India has approved a number of proposals and research

projects which include the installation of a pilot plant at Jamshedpur, Bihar, at an estimated cost of 1½ million rupees, to investigate the production of iron using non-coking coals by the low-shaft furnace process. An attempt will be made to replace coking coals in the iron-smelting industry in order to conserve coking coal reserves.

## JAPAN

### Ammonium sulphate

The Ammonium Sulphate Export Co., which was formed in August 1954 under the terms of the Fertiliser Supply and Demand Stabilisation Law, announced that the total contracts for export during the fertiliser year August 1954 to July 1955 would be 424,000 tons. This quantity had been brought for 9,300 million yen and would be sold for 9,100 million yen, making a deficit of 200 million yen, most of which was due to exports at a loss to Formosa.

## TUNISIA

### Uranium deposits

It has been announced in the press that as a result of investigations which have taken place during the past few years the presence of uranium in Tunisian phosphate deposits has been proved. The percentages of uranium present vary, but, taking an average of 20 g. in each ton of phosphates in an estimated deposit of 50 milliard tons, a total figure of something like 1 million tons of uranium has been arrived at.

## GREECE

### Oil refinery contract signed

The contract between the Greek Government and the German firms, Hydrocarbon, Krupp and Stahlunion, has finally been signed. According to reports, the refinery, which is to be erected on the coast near Elevis, will be ready in two years' time. Some 2,000 Greek workers will be employed on the project, which will cost \$2½ million. The capacity of the refinery will be 1.3 million tons p.a.

## ECUADOR

### Cement expansion

The country's second cement plant is reported to be in an advanced stage of construction at San Juan near Riobamba. Production is expected to start in November and to reach a rate of about 150 metric tons/day. The Government is also considering the construction of a third plant at Guapan in the province of Canar.

During 1954 the Guayaquil cement plant manufactured 94,000 metric tons of cement and about 10,000 metric tons were imported.

### Sir Harold Tempamy

*We record, with deep regret, the death on July 2 of Sir Harold Tempamy, C.M.G., C.B.E., F.R.I.C., who, up to March 1955 was editor of our associate journal World Crops. He was 73.*

*After a distinguished career as an agricultural scientist in the British Colonies, he returned to London in 1936 as the assistant to the Agricultural Adviser to the Secretary of State for the Colonies. Four years later he succeeded Sir Frank Stockdale as Agricultural Adviser. He retired from the service in 1946 and was knighted in that year.*

*It was in 1949 that he became associated with Leonard Hill Ltd., undertaking the editorship of World Crops which Mr. Leonard Hill founded in that year. Under his guidance, World Crops rapidly became a foremost authority on world agricultural problems, achieving an international circulation. Sir Harold's absence will be keenly felt in the agricultural world.*



## UNITED STATES

### New fertiliser plant

Thunderbird Chemicals Inc. are beginning the construction of a \$13-million fertiliser plant on a 122½-acre site near Kyrene, Arizona. Plans for the plant are being drawn up by Ebasco Services Inc., of New York. The plant, with the start of operations scheduled for about mid-1956, will manufacture anhydrous ammonia and other farm fertilisers, such as phosphates, sulphates and chemical urea.

### Synthetic ammonia process

The Koppers Co. Inc. has been granted North American rights to design, construct and license plants to use the L'Azote process for producing synthetic ammonia from coke-oven gas, refinery gases or any other source of hydrogen.

The process, which was first developed in Belgium, provides for more efficient utilisation of waste heat. Production of synthetic ammonia utilising the hydrogen in coke-oven gas is of special interest to the U.S. steel industry because it has experienced dwindling revenues from the sale of gas, due to the large increase in distribution and use of natural gas.

It is estimated that, if all the hydrogen available from coke-oven gas production in America was converted into synthetic ammonia, more than 11 million tons p.a. of synthetic ammonia could be produced. This would be about three times the target set by the Federal Government for production capacity in 1957.

## ISRAEL

### Copper project

It has been reported that equipment valued at \$3½ million for the erection of a copper plant at Timna, near Eilat, will be supplied from Germany

under the reparations agreement. The construction is expected to take from two to three years and America's Dow Chemical Corporation is understood to be taking part in its planning.

## FINLAND

### Leather industry's expansion

A recent survey reveals that Finnish leather production has risen in spite of competition from plastics and rubber. There are about 20 leather manufacturers in Finland. In 1954 the industry used 16,298 tons of raw skins and hides for tanning, compared with 14,660 tons in 1953.

Dr. Pekka Kontio, of Finland's State Institute for Technical Testing, points out in *Teknillisen Kemian Aikakauslehti* that the complex nature of leather has made its testing extremely difficult. Some of its properties can be tested scientifically, but in many cases only the old methods of testing by touch are available.

## BELGIUM

### Copper, sulphuric acid and uranium developments

Output of the Union Minière du Haut Katanga totalled, in 1954, 223,791 tons of copper against 214,116 tons in the previous year, 8,609 tons of cobalt against 8,278 previously, and 85,289 tons of roasted zinc concentrates against 62,809 tons in 1953. In 1954, the company produced 1,095 kilos of germanium oxide for the first time.

The subsidiary company, Sogechim, produced 30,826 kilos of cadmium sticks against 31,996 kilos in 1953. Output of sulphuric acid of this subsidiary is to be expanded to 110,000 tons this year against 83,512 tons last year.

A new roasting process for the treatment of cobaltiferous copper pyrites

is to be put into operation this year at the Shituru plant.

Uranium exports from the Shinkolomwe mine continued at satisfactory levels, while a process to produce uranium for use in atomic reactors is reported from the Societe Generale Metallurgique de Hoboken in Belgium.

## ARGENTINA

### Synthetic phenol

Farbenfabriken Bayer A.G. have been authorised to erect a plant at San Nicolas for the manufacture of synthetic phenol under the foreign investment law. The new venture will be known as Fabrica Argentina de Fenol y Derivados. Capital of 375,000 U.S. dollars will be supplied jointly by Farbenfabriken Bayer A.G. and the Argentine firm Bunge & Born. Local capital will be contributed by the Argentine concern Cia. Quimica S.A. Production planned is 1,800 tons p.a., sufficient to make Argentina independent of imports.

## SOUTH AFRICA

### Increased ammonia production

But for the increased output of nitric acid available from the new ammonia plant at Modderfontein, the uranium industry would have had to import substitute material at much higher cost. This was pointed out by Sir Ernest Oppenheimer, chairman of African Explosives and Chemical Industries Ltd., during his annual review. Dealing with the company's major plant extensions, he said that the main projects now in production are the first stage of the new ammonia plant (the second and third stages still being under construction), the fertiliser ammoniation and granulation plants at the Somerset West and Umbogintwini factories, and the first extension to the Klipspruit cyanide factory, the second extension being still under construction.

The new plants at Umbogintwini for the production of chlorine, caustic soda and associated products and for increasing sulphuric acid production are nearing completion and are due to start up within the next few months.

In view of the continually growing demands for nitrogen in both industry and agriculture, the company are at present engaged in planning further substantial extensions to its ammonia capacity. In the layout of the site of the new ammonia plant at Modderfontein, provision has been made for further extensions such as those now contemplated, Sir Ernest said.

The position and prospects of the subsidiary company, Rand Carbide Ltd., improved during last year.

## CHEMICAL & PROCESS ENGINEERING

### ENQUIRY BUREAU

Stratford House, 9 Eden Street, London, N.W.1

Readers requiring names of suppliers of raw materials, machinery or finished products should complete this form and attach it to their business note-heading. Please state approximate quantities required.

We wish to know names and addresses of suppliers of the following:

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